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FUNDAMENTAL AND APPLIED RESEARCH AND DEVELOPMENT IN METALLURGY

M. P. E. P.

Fabrication of Seamless Tungsten - 25 $^{w}/_{o}$ Rhenium Tubing

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Summary Technical Report to the
United States Atomic Energy Commission
for the period July 1, 1964 through June 30, 1965

S. Isserow, G. I. Friedman, R. G. Jenkins and A. L. Geary

July 15, 1965

Nuclear Metals

Division of Textron inc.

West Concord, Massachusetts

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UC-80 - Reactor Technology
(TID-4500 - 42nd Ed.)

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A. R. Kaufmann
Technical Director

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ABSTRACT

The development program was directed toward the establishment of extrusion and drawing techniques for tungsten - 25 w/o rhenium tubing in two sizes: 0.080-inch OD x 0.008-inch wall, and 0.250-inch OD x 0.020-inch wall. For each size, the primary extrusion is performed routinely. For the larger size, reliable techniques were developed and demonstrated for subjecting the extruded tubing to a series of moving-mandrel draws that iron the tubing to a reduction of about 30 percent. The successful application of moving-mandrel drawing and subsequent reeling for mandrel removal depends on the proper combination of mandrel material and working temperatures. In addition, the tubing must be properly conditioned by appropriate surface preparation and thermal treatment. For the smaller size, the proposed sequence is based on re-extrusion of the product of the primary extrusion. Although some apparently satisfactory tubing was obtained by re-extrusion, conditions for consistently obtaining sound tubing were not established. The billet for re-extrusion incorporates a removable core of calcium carbide within the inner molybdenum filler. This carbide core is removed chemically from the extruded tube by "acid drilling". The re-extruded tubing (OD about 0.1-inch) can be drawn with or without the inner extrusion fillers. The continuing metallurgical evaluations include some routine checks at various stages of fabrication as well as tests intended to guide the selection of anneals introduced in fabrication sequences. end

I. INTRODUCTION

This report describes accomplishments during the third year of the program in the development of fabrication techniques for tungsten-based tubing. Previous accomplishments on extrusion and drawing are reported in References 1 and 2.

Early in this year's program, it was agreed to concentrate on fabrication techniques for the tungsten - 25 $^{w}/_{o}$ rhenium. Accordingly, this report is limited to development work on this alloy. Earlier results or new experiments on unalloyed tungsten are introduced as they relate to problems with the alloy.

It was also agreed to concentrate on establishment of techniques for the following two sizes that were of particular interest to potential users:

	OD (in.)	Wall (in.)	Common Designation
A	0.080	0.008	80-8
B	0.250	0.020	1/4-20

In light of previous experience with the alloy,(2) sequences were formulated for the two sizes. The development program was thus devoted to demonstrating the various operations in the proposed sequences. These sequences have the following features: For both sizes, a powder metallurgy sleeve is first extruded. For the 80-8 tubing, the product of the primary extrusion is re-extruded. For both sizes, the final step is a series of draw passes, applied either to the doubly extruded material (for 80-8) or the singly extruded material (for 1/4-20). For the 80-8, drawing is intended primarily to reduce the outside diameter (sinking) and to improve its uniformity. For the 1/4-20, whose inside diameter must meet tighter dimensional tolerances, drawing also incorporates a hardened mandrel to reduce the wall thickness (ironing) and improve its uniformity.

Preparation of tubing in the two sizes thus involves combinations of some of the following operations: primary extrusion, secondary extrusion, drawing to sink the smaller tubing, and drawing to iron the larger tubing. Conditions for the primary extrusion were established the preceding year(2) and were applied rather routinely to obtain stock for processing to the two sizes. In addition, the drawing of small-diameter tubing to reduce and improve the outside diameter was accomplished without difficulty. However, considerable effort had to be devoted to the development of techniques for the other two operations: (1) re-extrusion to small-diameter tubing and (2) drawing of tubing of about 1/4-inch OD over a hardened mandrel. These two operations posed the main problems in the preparation of 80-8 and 1/4-20 tubing respectively, and are the principal subjects of this report. It will be seen that the drawing

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technique was established, permitting the fabrication of a total of 64 feet of tubing with high yield. In contrast, conditions have not yet been defined for re-extrusion.

The fabrication program continued to be guided by metallurgical evaluations of the materials at various stages. Sleeves were examined routinely. The examination of fabricated tubing was more experimental, being used to assess the effect of fabrication conditions or to guide the selection of new conditions such as for contemplated heat treatments.

The following report is organized on the basis of operations rather than tubing sizes. Separate sections describe the three phases of the program: extrusion, drawing and evaluation. These phases were respectively the responsibilities of G. I. Friedman, R. G. Jenkins and A. L. Geary. In each section, some of the detailed information (e.g., dimensions of billet components) has been omitted: this information is available in the monthly reports (NMI-2130 through-2139).

II. SUMMARY AND CONCLUSIONS

A sequence of extrusion and drawing was demonstrated for W - 25 w/o Re tubing with the following dimensions: 0.250-inch OD x 0.020-inch wall. Techniques of drawing and reeling had to be developed for obtaining this size from extruded stock with dimensions of about 0.29-inch OD x 0.030 inch wall. For the other size of interest (0.080-inch OD x 0.008-inch wall), the crucial step is re-extrusion of extruded stock with dimensions of about 0.38-inch OD x 0.040-inch wall. Considerable effort was devoted to this re-extrusion, but the proper conditions for consistently obtaining sound tubing were not defined. In line with the organization of this report on the basis of operations rather than tubing sizes, the extrusion and drawing operations are summarized separately. Some of the results of the metallurgical evaluations are also noted.

A. Extrusion

Primary extrusion was performed routinely by the filled-billet technique under conditions established previously. Sintered sleeves were thus converted into tube stock of two sizes, 0.29-inch OD x 0.030-inch wall and 0.38-inch OD x 0.040-inch wall; this stock was used for drawing and re-extrusion to smaller sizes. All the extrusion billets were preheated to 1600°C prior to extrusion with a glass lubricant through a zirconia-coated die. In most cases, the reduction was 16:1, but a reduction of 25:1 was also successful, especially when the ram speed was made high enough (about 250 ipm) to minimize loss of heat from the billet to the liner.

The proper conditions for consistently successful re-extrusion were not established. The search for these conditions was undoubtedly made more difficult by the incorporation of a calcium carbide core. Such a core is necessary to obtain a pilot hole which permits dissolution of the inner molybdenum filler at a practical rate. Calcium carbide is attractive because it is removable at a rapid rate by an "acid drill" consisting of a small-diameter tube connected to an acid reservoir. Conceivably, an alternative filler might perform more satisfactorily in re-extrusion while responding adequately to chemical removal. 4 py

With calcium carbide as the standard removable core, a combination of conditions was found that, at times, produced tubing that appeared to be satisfactory. The marginal nature of the process was shown by the failure of these conditions to give sound tubing consistently. The elusive factor(s) responsible for the inconsistent behavior could not be identified. It is clear, however, that better re-extrusions are obtained as a result of the following: a recrystallization anneal of the stock, and re-extrusion at a slow speed (about 50 ipm). The following conditions were fairly standard in the re-extrusions: molybdenum fillers, 1200°C billet temperature, and a reduction ratio of 16:1. The following changes did not appear beneficial: TZM fillers,

1500°C billet temperature, and a reduction ratio of 9:1. The possibility remains that an appropriate combination of filler material, temperature, and reduction ratio may be optimum and capable of giving satisfactory tubing consistently.

B. Drawing

Considerable progress was achieved in drawing the W - 25 w/o Re alloy by several methods. The combination of moving-mandrel drawing and reeling received the most attention, which led to the outstanding accomplishment, namely, establishment and application of a process sequence (Appendix I) for drawing 1/4-inch OD x 20-mil wall tubing from stock extruded to 0.29-inch OD x 30-mil wall. The successful sequence was achieved by (1) use of M-2 or M-3 steel mandrels hardened to R_c 60 to 65 and (2) adoption of 450 and 480°C respectively as the temperatures for the moving-mandrel drawing and the subsequent mandrel removal by reeling. For the 1/4-20 tubing, reductions of about 10 percent per pass were combined for a cumulative reduction of 30 percent.

Other methods of drawing were applied, but not as extensively as the moving-mandrel method. Drawing with a stationary mandrel (plug drawing) was applied successfully to larger extruded stock (0.38-inch OD x 40-mil wall); application of such drawing below about 0.31-inch OD x 30-mil wall was prevented by the very large draw forces, which caused tensile failure of the point or the drawn tubing -- and also deformation of the plug. Further reductions can be achieved by moving-mandrel drawing. Through such a combination of drawing techniques, the larger extruded stock was also converted to the desired 1/4-20, undergoing a cumulative reduction greater than 50 percent.

The route to 1/4-20 tubing may also incorporate sinking draws, i.e., draws involving changes in diameters with negligible changes in wall thickness. Such drawing was found most useful as a finishing step with minor dimensional changes after a series of moving-mandrel draws in the production of 1/4-20 tubing.

Drawing with a deformable mandrel, consisting of the molybdenum inner filler and generally also the graphite or CaC_2 core, was shown to be applicable in two steps in the path to tubing with 80-mil OD x 8-mil wall: (1) The product of the primary extrusion is rounded before being used for the re-extrusion; (2) the re-extruded tubing is drawn to the final diameter. In spite of the limited supply of re-extruded tubing, the last step was tested sufficiently to demonstrate that it can be applied with little difficulty. Diameter reductions of about 5 mils per pass can be combined for a cumulative reduction of about 20 mils.

Successful application of the drawing techniques requires attention to both the surface condition and the thermal history of the tubing. The initiation of microcracks at striations is avoided by surface conditioning,

incorporating centerless polishing with a belt and electropolishing. A stress-relief anneal for 1/2-hour at 1400°C after every draw was adopted, although it may not be essential after each pass. No improvement in drawing behavior resulted from a recrystallization anneal.

Some of the aspects of drawing developed earlier required little modification. Hot swaging provided the points; some difficulty was encountered in the small diameter range (about 0.1-inch). The graphitic lubricant was standard; additional attention had to be paid to its removal, which is particularly necessary when high-temperature anneals are frequent.

C. Evaluations

Metallurgical evaluations continued to guide the fabrication program. All the sleeves were checked. Evaluation of fabricated tubing was somewhat more limited than in the earlier work. Extruded tubes generally received a routine metallographic check. Some of the tubes also underwent ring compression tests to assess the effect of fabrication conditions. In addition, some tubes were subjected to experimental anneals, whose effects were assessed on the basis of metallography and ring compression tests.

The fifty extrusion sleeves (33 from the Bureau of Mines, 17 from a commercial source) were all satisfactory; no second-phase particles were found.

The properties of primary extrusions (all at 1600°C) were not affected by the following differences: source of sleeve, reduction of extruded size from the reference size of 3/8-inch OD x 40-mil wall to 1/4-inch OD x 20-mil wall (or a somewhat larger size to be drawn to 1/4-20), inclusion of graphite core, and increase of the reduction ratio from 16:1 to 25:1. Annealing studies led to the recommendation of an anneal of 15 minutes at 1800°C to obtain fully recrystallized, relatively fine-grained stock for drawing. The various as-extruded tubes appeared quite free of second phase. Some second-phase particles were seen in material annealed at 1800°C.

Re-extruded tubing is fully recrystallized by a one-hour anneal at 1600°C. An annealing temperature of 1700°C is required for an equiaxed structure.

III. EXTRUSION

The extrusions were of two distinct types (primary and secondary) which are discussed separately. The primary extrusions applied the filled-billet technique to convert sintered sleeves, prepared by powder metallurgy, into tubing with diameters near 0.3-inch. These extrusions were performed rather routinely and provided stock for drawing or secondary extrusion (re-extrusion). The re-extrusions sought to reapply the filled-billet technique to convert the product of the primary extrusion into tubing with diameters near 0.1-inch. Considerable effort was devoted to definition of proper conditions for successful re-extrusion.

A. Primary Extrusions

The primary extrusions of W - 25 $^{w}/o$ Re demonstrated the feasibility of extruding tubing in a size range below that which is feasible by standard mandrel techniques, provided material for drawing to 1/4-inch OD x 0.020-inch wall, and supplied tubing stock for the re-extrusion phase of the program.

The tubes produced were all extruded by the "filled-billet" technique, under conditions that are described in detail in Ref. 2. In brief, the process entails fitting a graphite-cored molybdenum rod inside a W-Re tube blank and encasing the whole in a thick-walled outer molybdenum jacket. The pressed-and-sintered molybdenum jacket has an outside diameter of three inches, with an inside diameter approximately three mils larger than the W - 25 $^{w}/o$ pre-extrusion sleeve. For the 1-1/2-inch OD sleeves used to provide re-extrusion stock, this arrangement gives a 3/4-inch outer wall of molybdenum in the billet, while, for the 1.1-inch sleeves used for the 1/4-inch tubes, the molybdenum jacket is nearly one inch thick. There is also a 3-mil clearance between the sleeve and the molybdenum inner filler. The graphite core* is either 0.5- or 0.6-inch in diameter. The billet is heated to 1600^{o}C and extruded at a 16:1 reduction **, using glass as a lubricant, through a zirconia-coated steel die. The zirconia is removed, and the die is recoated after each extrusion. Data on extrusion conditions, extruded tube dimensions, and the use to which the tubes were put are to be found in Tables 1 and 2. Dimensions of the individual billet components are available in the respective monthly reports and are not reproduced here.

Midway through the program, the extrusion speed was increased from 100 to 250 ipm. This increase was intended to reduce the heat loss

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* High-Density Mold-Grade Graphite

** Billet 189, containing a 1.86-inch OD Bureau of Mines arc-cast sleeve, was extruded at a 25:1 reduction from the 3.5-inch liner. Billets 200, 201, 202, 236 and 237 were also extruded at 25:1 but from the 3-inch liner.

from the billet to the liner during extrusion, thereby lowering the extrusion forces and consequent washing of the die surfaces. This change was initiated after an attempted 25:1 reduction of Billet 201 at 100 ipm stalled the press. The following billet, No. 202, identical in all respects to No. 201, was successfully extruded at 250 ipm. The success of this approach may be seen from the fact that Tube 207 required 510 tons for extrusion at 100 ipm, whereas Tubes 242-245, formed from similar billets under otherwise identical extrusion conditions, required an average force of 410 tons. All primary billets subsequent to Billet 201, with the exception of Billet 207, noted above, were extruded at 250 ipm or higher.

It appeared that outgassed graphite cores might reduce interstitial contamination of the tube blank during billet heating. Some tubes (Nos. 226-230, 240, 241) were, therefore, extruded from billets containing graphite cores that had been subjected to an 1800°C vacuum heat treatment. Because of the difficulty encountered in drilling the cores from the first five of these tubes, the graphite heat treatment was later discontinued. No analyses of interstitial contaminants have been performed for any of the primary extrusions.

Except for four tubes, all primary extrusions fell into two categories. In the first group, tube blanks with a 1.136-inch OD and a 0.100-inch wall were extruded to approximately 0.290-inch OD x 30-mil wall. (It is typical in a filled-billet extrusion of W - 25 w/o Re for the tube blank to undergo only 95 to 97% of the reduction indicated by the press tooling.) From this size, the tubes were drawn to 1/4-inch OD x 0.020 wall.

In order to increase the yield of usable tubing obtainable from the extrusion of a W - 25 w/o Re sleeve, the sleeve length for the 1/4-inch tubes was doubled from four to eight inches. The first of these longer sleeves (Nos. 208,209) were of thinner wall than specified, due to initial fabrication difficulties on the part of the sleeve supplier.

The second group of tubes was fabricated from tube blanks with an OD of 1.500 inches and a 0.160-inch wall; these were extruded to approximately 0.380-inch OD. Some were subsequently drawn to 0.375-inch, the size selected for the re-extrusion development work. A line of knobs and depressions found on the surface of Tube 238 is attributed to damage to the sleeve which occurred when it became necessary to press it into place in the billet, after it had become jammed in the molybdenum outer filler.

The four other primary extrusions were performed either as pilot runs to provide information necessary for the extrusion of tubing in the two categories mentioned above, to test the feasibility of the filled-billet technique for tube blank sizes other than in the two

main categories and at higher reduction ratios, or to provide tubing for other installations (Tubes 236 and 237).

The net result of the extrusion effort described above has been to demonstrate the ability to extrude primary W - 25 $^{w}/_{o}$ Re tubes routinely. Further work in this area could involve the use of a soluble core, such as calcium carbide, in the center of the billet. Such a core could be smaller than the graphite core presently used, and would still be large enough to permit rapid core dissolution (see "acid drill", Section B Part 2, p. 10.)

In addition, the use of finer-grained sleeves and finer-grained (at the extrusion temperature) fillers should be explored in an attempt to improve the surface quality of the extruded tubing.

B. Secondary Extrusions (Re-extrusions)

1. Introduction and Objective

The major part of the experimental effort during this year's extrusion work was devoted to the search for a suitable technique for re-extruding the 3/8-inch W - 25 $^{w}/_{o}$ Re tubing (produced as described above) to approximately 0.090-inch OD x 0.009-inch wall. Much of the work in the program, including the final set of five re-extrusions under identical conditions, was characterized by apparent successes which could not be reproduced consistently. Many factors, i.e., reduction ratio, extrusion temperature, core size, billet-filler material, condition of the W-Re tube stock, and extrusion speed, were investigated. The following factors are believed to be particularly important: extrusion temperature (1200°C), extrusion speed (no greater than 60 ipm), and condition of the stock tube. (The W - 25 $^{w}/_{o}$ Re must be in the recrystallized state.) Even with the recognition of these factors, the process is not yet fully defined, and further work remains to be done before fully sound tubing can be produced on a routine basis.

The experimental effort detailed below involved an investigation into the following factors with respect to their significance in re-extrusion:

(1) Determination of maximum reduction (Section 3.a.)
(2) Incorporation of a soluble core (Section 3.b.)
(3) Use of a stress-relief anneal (Section 3.c.)
(4) Comparison of effect of stress-relief, reduction and temperature on identical stock tubes (Section 3.d.)
(5) Relationship between tube and die size, reduction, and recrystallization anneal (Section 3.e.)
(6) Extrusion speed and use of stiffer fillers (Section 3.h.)

Data on extrusion conditions and on the dimensions of stock tubes and extruded tubing are to be found in Tables 3 and 4. Dimensions of

the individual billet components are available in the respective monthly reports and are not reproduced here.

2. General Experimental Technique

The billet design and extrusion procedure used for the re-extrusions followed the general pattern developed in last year's work. All extrusions were based on a filled-billet design, which in the case of the large majority of the tubes consisted of a calcium-carbide-cored molybdenum inner filler, with a steel-jacketed molybdenum outer filler.

In the center of the billet, inside the W - 25 $^{w}/o$ Re stock tube, was a 290-mil inner filler of molybdenum or molybdenum with a calcium carbide core (see below, Section b., p. 11.)

In the case of billets extruded from the 3-inch liner at or below 1235°C, the 3/8-inch W-Re stock tube was inserted into a drilled hole in a 2-inch OD pressed-and-sintered molybdenum bar. This outer filler was, in turn, fitted into a 2.8-inch steel can, which was evacuated and sealed*. Just prior to extrusion, a 16-gauge spun-copper can, heated to 900°F, was inserted into the press liner. As the press ram advanced, the billet was pushed into this open-ended can, which melted during the course of the extrusion.

All billets extruded at temperatures above 1235°C had all-molybdenum outer fillers 3 inches in diameter. With a small number of exceptions, all billets were extruded from a 3.05-inch diameter liner in the 1400-ton extrusion press. The reasons for choosing this liner size and press are discussed below.

Billets extruded at 1200°C are heated in stainless steel or graphite muffles in an atmosphere of pre-heated, flowing argon inside a resistance-heated box furnace. The temperature of these billets is continuously monitored by an Inconel-702-clad, swaged, Chromel-Alumel thermocouple adjacent to the billets inside the muffle. The calibration of this thermocouple is periodically checked. Billets heated in the resistance furnace are manually removed from the muffle and are either carried directly to the loading cradle, or are allowed to roll down the bare glassing table onto the loader. In either case, the billet transfer time is roughly comparable to that achieved with the induction-heated billets.

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* For the 2-inch liner, the molybdenum diameter was 1.5-inch and the steel diameter 1.8-inch.

Billets extruded at temperatures above 1235°C were heated by induction. The induction furnace is described in detail in Ref. 2, p. 9, and in Figure 9 . Billet temperatures are measured by sighting with an optical pyrometer through a glass port in the top lid of the furnace. The pyrometer is focused on the bottom of a 1-1/2-inch deep hole drilled into the rear end of the billet's molybdenum tail plug. When a trap door at the bottom of the furnace is sprung, the billet drops down through a chute onto a glassing table, and from there rolls onto the press loading cradle, which automatically raises it to a position in line with the press ram. Less than ten seconds elapse from the time the billet drops from the furnace to the moment the ram commences its advance.

All billets are extruded directly into a Transite-lined steel catch tube, where they cool slowly to a black heat before being removed. By cooling the tubes in this manner, a high degree of straightness is achieved.

After extrusion, the outer fillers are dissolved in acid: 50 $^{v}/o$ conc. nitric acid - 50 $^{v}/o$ water for the steel, and 40 $^{v}/o$ conc. nitric acid - 20 $^{v}/o$ conc. sulphuric acid - 40 $^{v}/o$ water for the molybdenum. The tube core is then removed with an "acid drill" if the extruded tube appears sound. This drill consists of a small-bore tantalum tube, which is mounted in a nylon block through which it is connected to a hydrochloric acid reservoir. The drill is positioned vertically so that acid issues forth from the open end at the top. The extruded tube is placed in axial alignment with the drill, with the calcium carbide core resting on the end of the drill tube. In this way, the entire calcium carbide core is dissolved as the extruded tube drops, at a rate of approximately two feet per hour. With the core removed, the nitric-sulphuric acid solution is flushed through the tube to dissolve the inner molybdenum filler. For those tubes which are to be deformable-mandrel drawn after extrusion, the core may be drilled out at any stage in the drawing sequence (see p.32).

All tubes were visually inspected for defects after the outer molybdenum filler had been removed. The tubes were then measured, and those tubes that appeared sound had their inner fillers removed. They were then tested for leaks with the He mass spectrometer and subjected to a fluorescent dye penetrant test. Radiographic and eddy current inspections were used for some of the tubes.

3. Development of the Fabrication Process

a. Determination of Maximum Reduction

Last year's work on this program had shown that it was possible to re-extrude 3/8-inch tungsten - 25 $^{w}/o$ rhenium tubing with a solid molybdenum inner filler at a 9:1 reduction. This year's work

began with a determination of the approximate limit, i.e., greatest reduction, to which this process could be extended by re-extruding tungsten and tungsten - 25 $^{w}/o$ rhenium at 25:1 and 36:1 reductions with solid molybdenum inner fillers.

The first re-extrusions were performed in the 2.04-inch liner of the 300-ton press. Although the 25:1 extrusion of both the tungsten (Tube 180) and the tungsten-rhenium (Tube 182) were completed successfully, the attempt to re-extrude tungsten at a 36:1 reduction resulted in a press stall (Billet 181). Consequently, the W - 25 $^{w}/o$ Re billet (No. 187) was modified (by adding an additional 1/2-inch of steel to the outer can) in such a way as to "dilute" the billet and reduce the fraction of molybdenum in the billet. In this way, it was possible to reduce the extrusion pressure without decreasing the ratio of molybdenum to tungsten-rhenium in the billet. Since forces larger than 300 tons were now necessary, operations were transferred to the 3.05-inch liner in the 1400-ton press. It should be noted that, whereas the smaller press has a maximum extrusion speed of 65 ipm, the larger press is capable of operating at speeds ranging from approximately 15 ipm to more than 1500 ipm. A speed of 100 ipm was selected for the re-extrusion work.

Billet 186, the first W - 25 $^{w}/o$ Re re-extrusion in the large press, was successfully extruded at a reduction of 25:1. Although Billet 187 extruded smoothly at a presumed 36:1 reduction, it was found that the tube contained chevron-shaped tears (Figure 1 shows such tears in other tubes). The tears were attributed to the high reduction that the tube had undergone. While the press tooling dictated a nominal 36:1 reduction ($R = \frac{\text{Liner Area}}{\text{Die Area}}$), measurements of the extruded tube, compared with the starting stock tube size, indicated a reduction of approximately 45:1.

With this indication of the limiting reduction, it was decided to concentrate on re-extrusion in the range of 16:1 to 25:1 reductions, which was considered adequate for obtaining the desired final size from the 3/8-inch starting stock.

b. <u>Incorporation of a Soluble Core</u>

At this point, consideration was given to developing an alternative to the solid molybdenum inner filler that remained inside the extruded tube. It was found that the mixed acids used with the acid drill would dissolve this inner filler, but at a rate of approximately three-eighths of a linear inch per hour. Earlier work on another program had demonstrated the feasibility of coextruding an easily-dissolved substance, such as calcium carbide, as the innermost core of a tungsten filled-billet extrusion. It was therefore decided to investigate the possibility that a calcium carbide core could be incorporated into the re-extrusion billet.

Three billets, Nos. 190, 191 and 192, were prepared with holes 201, 221 and 250 mils in diameter drilled down the center of the inner molybdenum filler. Into these holes calcium carbide powder, -20 +30 mesh, was tamped. The billets were extruded through a 20:1 reduction at 1200°C. Although it would have been gratifying if all three extrusions had been successful, it was not too disappointing to learn that only Tube 190, containing the least CaC_2, was satisfactory, for this seemed only to indicate that, in the other two tubes, the limit of a still undefined CaC_2:Mo ratio had been exceeded.

Since Tube 190 had actually undergone a total reduction of 23.5:1, i.e., 12 percent greater than that to be expected from the press tooling, it was considered that the tamped powder of approximately 50 percent theoretical density did not offer sufficient support to the inner molybdenum filler. This could be remedied by pre-extruding calcium carbide in a molybdenum jacket, thus densifying the powder, as well as putting it into an easily-used form. Therefore, a steel-clad molybdenum billet with a calcium carbide core was extruded at 1200°C at a reduction of 16:1. From this rod, calcium carbide-cored molybdenum inner fillers were machined.

Prior to Billet 205, stock tubes were used in the as-extruded condition. In order to obtain more consistent component dimensions with closer fit, the stock tubes for Billet 205 and all subsequent billets were deformable-mandrel drawn to an outside diameter of 0.375-inch.

Billets 205 and 206, containing pre-extruded calcium carbide cores, were assembled and extruded in order to confirm and improve upon the results of Tube 190. The stock consisted of a section of Tube 194, which had been deformable-mandrel drawn. A 19.4:1 reduction ratio was used for Billet 205 in order to obtain a tube closer in size to the desired 80-mil diameter. Both of these tubes were failures; i.e., Tube 205 consisted of six pieces, while No. 206 was in three pieces.

c. Use of Stress-Relief Anneal

To test the possibility that a stress-relief anneal would eliminate failure of drawn stock, two pieces of tubing drawn from Tube 194 were subjected to a 1450°C vacuum anneal. One piece of tubing had as an inner filler CaC_2 tamped into a 0.20-inch drilled hole, while the other utilized pre-extruded CaC_2. These two tubes, Nos. 209 and 210, were also failures, despite the fact that Tube 209 was a virtual twin to the successful Tube 190. The one difference between the two tubes is that Tube 190 originated from stock Tube 178, which was in the as-extruded state, whereas stock Tube 194, in the drawn-plus-stress-relieved condition, was used for Tube 209.

d. Comparison of the Effect of Stress-Relief Heat Treatments, Reduction Ratio and Extrusion Temperature on Identical Stock Tubes

A series of extrusions was planned in which all billets would contain sections of the same stock tube (T199), with sections of the same pre-extruded CaC_2 as inner fillers. The objective in this series of extrusions was to determine what, if any, stress-relief treatment would be required to ensure the extrusion of sound tubing. At the same time, billets were to be extruded at a higher temperature or a lower reduction respectively, to see whether either change provided better re-extrusion conditions. In this series, Billet 212 was the control, containing an as-extruded-and-drawn section of stock Tube 199. This billet was extruded at 1200°C at a 16:1 reduction, duplicating Tube 190.

Billets 213 and 214 differed from Billet 212 in that the stock tube sections used were stress relieved at 1350 and 1450°C, respectively. A solid molybdenum outer filler was used for Billet 215 which was to be extruded at 1300°C. Billet 216 was of standard steel-plus-molybdenum design, but was to be extruded at a 9:1 reduction, rather than 16 or 20:1.

Billets 212, 213 and 214 produced torn and broken tubes. Billet 215 stalled at 1300°C; the outside was machined to fit the liner and extruded at 1500°C, producing a short, sound piece of tubing, which had undergone a total reduction of over 30:1. Tube 216, the 9:1 extrusion, was also satisfactory, although of larger diameter and greater wall thickness than usual, because of the lower reduction ratio.

The results of this series of extrusions indicated that:

(1) A stress-relief anneal of the stock tube is not sufficient to provide good tubing.

(2) Extruding at an elevated temperature, such as 1500°C, might be of some advantage.

(3) Extruding at a lower reduction, i.e., 9:1 rather than 16:1, would possibly be necessary for 1200°C re-extrusions.

e. Examination of the Relationship between Tube and Die Size, Further Exploration of Reduction Ratio and the Use of a Recrystallization Anneal

Based on the results of the preceding group of extrusions, it was decided to assemble a series of billets that would test the effectiveness of: a) a lower reduction ratio on smaller stock tubes, and b) a recrystallization anneal. An additional billet was used to test the hypothesis that the absolute difference in size between

the stock tube and the extrusion die is a controlling factor. Billet 220 was therefore extruded through a 0.510-inch die in the 2.04-inch liner on the 300-ton press for the 16:1 reduction.

Billets 221 and 222 were to be extruded through a 9:1 reduction at 1500 and 1200°C respectively, using a smaller stock tube size (0.284-inch) rather than the usual 0.375-inch, so that their as-extruded size would be comparable to that resulting from the 16:1 re-extrusions. The stock tubes for Billets 223 and 224 were subjected to a one-hour, 1800°C recrystallization anneal and were extruded at 1200 and 1500°C respectively. Billet 225 was also extruded at 1500°C but, in this case, the stock tube was in the extruded-plus-drawn state. Stock Tube 217 was used for Billet 225, whereas the three other 16:1 extrusions used stock Tube 199.

Billet 220, extruded through the 0.510-inch die, resulted in a fragmented tube. Tubes 221 and 222, for which 9:1 reductions were planned, actually went through 14.4:1 (based on the press tooling) reductions, due to the use of an incorrect die (0.805 rather than 1.02 inches). Both of these tubes (extruded at 1500 and 1200°C) were fragmented. Tubes 223 and 224, extruded from recrystallized stock tubing, were of good quality, while Tube 225, the 1500°C extrusion of a non-heat-treated stock tube, contained many tears.

This series of extrusions indicated that a high-temperature anneal is a prerequisite for re-extruding W - 25 $^{w}/o$ Re, for the stock tubes extruded without this treatment (Tubes 220, 221, 222, 225) at both 1200 and 1500°C, were failures, whereas Tubes 223 and 224, extruded from annealed stock tube sections, were both successful.

f. Selection of Optimum Extrusion Temperature and Reduction Ratio

The next step in the investigation was to determine whether 1200 or 1500°C was the more favorable temperature for re-extruding the recrystallized tubing; at the same time, we wished to determine whether a 19.4:1 reduction would also be successful.

Accordingly, Billets 232 and 233 were extruded at 1200°C, through reductions of 16 and 20:1; Billets 234 and 235 were extruded at 1500°C, at the same respective reductions. Not one of these four extrusions was a complete success, although there were sections 12 to 55 inches long in different tubes that appeared sound.

g. Re-Examination of Extrusion Variables and their Relation to Tube Stock

After the discouraging results of these last four extrusions, it was decided to re-examine the various factors explored up to this point, and to consider their relation to the history of the tube

stock. It was suspected that perhaps some stock tubes were capable of being re-extruded while others, because of surface conditions, small differences in primary extrusion conditions, or other factors, were unsuitable for re-extrusion.

In addition, consideration was given to the metallurgical condition and stiffness of the molybdenum inner filler. It was therefore decided to replace the molybdenum shell on the pre-extruded Mo-CaC_2 inner fillers with virgin pressed-and-sintered molybdenum in some billets, and with TZM in one other. It was also considered possible that the 5-minute "soaking" time for the induction-heated 1500^{o}C billets was inadequate, and so one billet would be held at temperature twice as long as before. To investigate these points, six tandem billets, each containing annealed stock tubes from more than one primary extrusion, were prepared (Table 3, T246-T251).

Billet 246, containing sections of stock Tubes 217 and 199, was extruded through a 16:1 reduction at 1200^{o}C, and served as a control.

In Billets 247-251, the molybdenum for the pre-extruded Mo-CaC_2 inner filler was removed and replaced by virgin pressed-and-sintered molybdenum. Billet 247 had this virgin-molybdenum inner filler as the only change. It contained stock Tubes 217 and 239. Billets 248 and 249 were extruded through 9:1 reductions. Billet 248 contained the same 3/8-inch stock tubes as Billet 247, while Billet 249 contained 310-mil stock tubes, so that these extruded tubes would compare in size to the 16:1 reductions. Billet 250, containing one section of Tube 217 annealed at a commercial facility and two sections of the same tube annealed at this laboratory, was extruded at 1500^{o}C; the time at temperature in the induction furnace was doubled from 5 to 10 minutes. Billet 251 was identical to No. 247, except that the pre-extruded molybdenum in the Mo-CaC_2 inner filler was replaced by wrought TZM.

The results of this series were disappointing. No one billet or stock tube resulted in completely sound tubing. As shown in Table 3, the front section of Billet 246, the center section of No. 250, and the rear section of Billets 247, 250 and 251 produced sound tubing. No combination of conditions consistently gave sound tubing. Thus, the series was inconclusive in that it did not permit specification of conditions for obtaining sound tubing. None of the following was demonstrated to be a sufficient condition: the use of virgin molybdenum or TZM to replace the pre-extruded molybdenum, a lower reduction ratio, the use of a longer heating time for the 1500^{o}C extrusion, or the use of different tube stock.

h. Extrusion Speed and Use of Stiffer Fillers

Since primary tubes were successfully extruded at speeds near 400 ipm, no consideration had previously been given to the possibility that the coextrusion was sensitive to speed. (Compare extrusion of tungsten with all-steel fillers, Ref. 2, p. 23 .) With one exception (Tube 220), all re-extrusions following Tube 182 had been performed in the 1400-ton press at speeds of at least 90 ipm; in contrast, Tube 182, which was sound, had a solid molybdenum core and had been extruded at the maximum speed of the 300-ton press, 65 ipm. It was therefore decided to run a series of extrusions at reduced speed. Also, TZM inner and outer fillers would be tried, since it was felt that this alloy, being stiffer than unalloyed molybdenum, might better match the flow characteristics of the W-Re at extrusion temperatures. These and all the subsequent extrusions contained tamped CaC_2 and recrystallized W - 25 $^w/o$ Re (except for Billet 259; see below).

Billets 252, 253, 254 and 255 had TZM inner and outer fillers. Billets 252 and 253, to be extruded at 1200°C, had steel outer cans, while Billets 254 and 255, 1500°C extrusions, had molybdenum outer cans. Billet 256, serving as a slow-speed control, was made up of the standard molybdenum and steel components for a 1200°C extrusion. All billets contained tamped CaC_2 cores.

Tube 252, extruded at 50 ipm, erupted from the die. The other 1200°C - TZM billet, No. 253, extruded at 100 ipm, also came from the die in short lengths. The remaining billets extruded satisfactorily. Post-extrusion processing revealed that (a) Billet 254, the 1500°C, 100-ipm, TZM-filled billet, resulted in a severely undulating tube; (b) Tube 255, (1500°C, 50 ipm, TZM) was of better quality, but still had a somewhat bumpy surface, and (c) Tube 256 (1200°C, 50 ipm, Mo) appeared sound. At this point, the low speed appeared beneficial, at least with molybdenum at 1200°C. TZM offered some promise at 1500°C. It was decided to duplicate Tubes 255 and 256, with the only change being a slightly larger CaC_2 core (200, instead of 172 mils), since there had been some difficulty in acid drilling the core from Tube 255. In addition, one molybdenum-filled billet, No. 259, was prepared for 1200°C extrusion from the 2.04-inch liner in the 300-ton press; the stock tube in this billet was not annealed.

Tube 257, duplicating Tube 256 (1200°C, 50 ipm) was sound, with a slightly undulating surface. Tube 258 (1550°C*, 50 ipm) was less

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* For some of the billets (see Table 4) the temperature was increased to 1235 or 1550°C to compensate for greater heat losses to the tools in a slower extrusion.

satisfactory than Tube 255, possibly because the extrusion was begun at a speed of 90 ipm and only gradually decreased to 45 ipm. Billet 259, containing the as-extruded stock tube extruded at 50 ipm in the smaller press, resulted in a three-piece tube.

To complete the test of the different combinations of filler material, temperature and speed, two more billets containing recrystallized W - 25 $^{w}/o$ Re were extruded at low speed. Billets 260 and 261 used molybdenum at 1550 and 1235^{o}C respectively. Billet 260 produced a poor tube. Billet 261 duplicated Billets 256 and 257 on the small press and was fairly successful. A small dimple on the surface of this tube is tentatively attributed to unevenness in the tamped calcium carbide core, or the possibility that at this spot calcium oxide or carbonate was present immediately before the depression. The same batch of calcium carbide was used throughout the program, and the possibility exists that, even though the container was kept tightly closed, the carbide reacted with atmospheric moisture to form one or both of these other substances, differing in deformation behavior from the carbide. Presumably, a pre-extruded CaC_2 core would deform more uniformly.

From this series, it was concluded that the following are required: recrystallized W - 25 $^{w}/o$ Re, Mo fillers, 1200^{o}C, and a speed no greater than about 60 ipm.* It was subsequently found, however, that although the above conditions are necessary, they are not completely sufficient to insure sound tubing. Five billets, Nos 262-266, containing molybdenum fillers, were extruded at 1200^{o}C, 50-60 ipm, with only fair results. Three of these tubes contained one or more chevron tears; the remaining two (T265 and T266), while of good over-all quality, each had a localized region where the tube underwent an abrupt change in diameter. These results show that further work is required in order to define completely the conditions for the consistently satisfactory re-extrusion of W - 25 $^{w}/o$ Re. Further work should also seek improvements through the return to pre-extruded CaC_2 and application of a milder recrystallization anneal. For whatever it may be worth, attention is called to the fact that the best tubes in the final sets, starting with T252, were derived from the primary extrusion T239.

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* A corollary result of the lower extrusion speed is the greater reduction that takes place in the tube dimensions. Whereas tubes from billets extruded at a speed of 90 ipm undergo reductions less than nominal, the slower extrusions produce tubes that are smaller than predicted from the press tooling. For these nominal 16:1 reductions, the typical reductions actually undergone by the tubes are 15.6:1 at the higher speed, and 17.5:1 at the lower speed.

IV. DRAWING

A. Introduction

Considerable progress was achieved this year in the drawing of tungsten - 25 $^{w}/o$ rhenium tubing. The previous year's report (NMI-1264) described the development of procedures for drawing tungsten at 580°C and W - 25 Re at 510 or 580°C. Plug drawing had successfully produced sound W - 25 Re tubing with outside diameter nominally 0.3-inch and wall nominally 23 mils after total reductions of approximately 40%. Moving-mandrel drawing, although applicable, was hampered by difficulties with mandrel removal and for this reason was not emphasized. Drawing with the inner molybdenum extrusion filler acting as a deformable mandrel and drawing without a mandrel had been successfully used in limited tests with the alloy. The rhenium alloy extruded at temperatures from 1500°C to 2000°C required no predraw anneal and seemed to require no stress-relief anneals during drawing. Although the alloy was less susceptible to cracking than the unalloyed tungsten it required much higher draw forces and was plagued by deformation of the mandrel during drawing.

Recent work has been primarily concerned with developing processes capable of producing W - 25 Re tubing with specific dimensions in two sizes:

(1) 1/4-inch OD with a 20-mil wall

(2) 0.08-inch OD with an 8-mil wall.

These sizes are referred to as 1/4-20 and 80-8 respectively in this report.

Pilot production runs were to be made to determine the reliability of the processes and to provide material for use elsewhere. Additional tubing was sized to special dimensions for use in the re-extrusion portion of the program (p. 8) and for evaluation or irradiation at other sites.

The drawing section of the present report will describe the processes developed, their capabilities and limitations. Problem areas and their cures, if found, will be discussed using references to specific draw tests. Pilot production of 1/4-20 tubes is covered in the epilogue.*

B. Equipment

1. General

Most of the equipment used during this program was the same as that described for the earlier programs in NMI-1260 and NMI-1264. New drawing dies and mandrels were procured for the present program.

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*After the process had been developed under Contract No. AT(30-1)-2784, the pilot production of 1/4-20 tubing was undertaken for the CANEL operation of Pratt and Whitney. This pilot production is the subject of the epilogue (p. 43).

2. New Dies

Suitable dies similar to the design shown in Figure 18 , NMI-1264, were made from either 97 WC - 3 Co or 94 WC - 6 Co cemented carbide nibs. These nibs were placed in hard M-2 or T-1 steel casing (R_C 46-50) designed to provide an interference fit at 580°C, the nominal "use" temperature of the dies at the time of the order. The standard nib design incorporated a standard entry bell which blended with an 18° entry angle which, in turn, blended with a bearing surface with a length equibalent to about 15 percent of the hole diameter. The back relief was a minimum of 3/32-inch long with a 30° angle.

3. New Mandrels

All new mandrels were obtained in the hardened and ground condition from the following sources:

(1) H-13 - Matt J. McDonald Co., Boston, Mass.

(2) M-3 - " " " " " "

(3) H-12 - Atlas Alloys Co., Cleveland, Ohio (Welland, Ontario, Canada).

(4) Lescalloy-718 - Latrobe Steel Co., Latrobe, Pennsylvania

(5) M-2 - ACE Drill Co., Adrian, Michigan

(6) NM-100 - Nuclear Metals Division of Textron Inc., West Concord, Mass.

Intermediate sizes were obtained by centerless grinding of the as-received material. All mandrels were oxidized by heating to 425 to 450°C before use.

C. Auxiliary Operations

1. Surface Preparation

Tungsten - 25 $^{w}/o$ rhenium tubing in the as-extruded condition generally had pronounced surface striations aligned with the extrusion direction. Moreover, there was uncertainty that all of the inner molybdenum extrusion filler had been removed. These conditions led to incorporation of three steps into the procedure for preparing the extruded tubing for drawing. Beginning with Series 154 the procedure for preparing tubes for drawing incorporated, with few exceptions, the following surface treatments after the H_2SO_4 - HNO_3 pickling step:

(1) Hydrogen peroxide "over-pickling" of inner surface.

(2) Centerless abrasive belt polishing of outer surfaces.

(3) Electropolishing of all surfaces.

The first step was intended to remove any residual molybdenum. The other two steps made the surface smoother than as extruded.

Hydrogen peroxide removed traces of molybdenum remaining on the interior surface (30% H_2O_2 attacks molybdenum and to a lesser extent the W-Re). Prior to the introduction of the H_2O_2 step, traces of molybdenum were carried to the end of at least one draw series (Series 139) presumably because the molybdenum became passivated towards the end of the H_2SO_4 - HNO_3 pickling. Since absence of the molybdenum could not be satisfactorily ascertained after the H_2SO_4 - HNO_3 treatment, it seemed expedient to overpickle the interior to ensure that the molybdenum had been removed. In practice the tube was plugged with rubber at one end and, in or near a vertical position, was filled with 30% H_2O_2 for 1/2 hour. The plug was then removed and the tube was drained and rinsed with tap water.

Mushrooming (i.e. upsetting) of the as-extruded striation peaks during drawing resulted in lubricant entrapment (e.g. Tests 134 and 139). Moreover, much of the micro and macro longitudinal cracking observed during drawing (see p. 42) may have resulted from excessive stress concentrations at the striations. In order to prevent lubricant entrapment and reduce sites for stress concentrations as well as improve the surface of the finished tubing, predraw surface smoothing by centerless polishing and/or electropolishing was made a part of the process.

Centerless polishing was the first step towards smoothing the outer surface. Centerless polishing using 50-, 80-, and 120-grit aluminum oxide or 320-grit silicon carbide belts nearly eliminates the striated appearance. Subsequent reappearance of the striations after electropolishing indicates that the W - 25 Re smeared considerably during the abrasive polishing step. The final outer surface was nevertheless much smoother than the as-extruded surface. The bulk of the polishing was done with 120-grit aluminum oxide or 320-grit silicon carbide belts; the coarser belts (50- and 80-grit) produced fairly deep circumferential scratches. Three passes were generally sufficient to remove about 1 mil from the surface. Optimum conditions were not established. It was noted that the W - 25 Re wore the belts quite rapidly and did not respond as would conventional metals to micrometer adjustment of the position of the pressure roll which normally permits controlled metal removal.

Electropolishing required different techniques for the outer and inner surfaces. The outer surfaces used the semi-cylindrical stainless steel (Type 304) cathode and the techniques in NMI-1264, p. 40 . The internal polishing procedures were first checked with tungsten tubing or W - 25 Re tubing of inferior quality, notably Tube 193, which was very nonuniform as a result of die wash. Because of the extreme difficulties

inherent in mechanical smoothing of small-bore interiors, internal electropolishing was used. The anodic electropolish technique used an aqueous solution of NaOH (20 g. NaOH/liter) in a vertical glass tube and a tubular steel cathode inserted within the W-Re tube. The cathode, a few inches longer than the W-Re tube, did not quite extend to the bottom end of the vertical W-Re tube; the inside of the steel tube served as an inlet for air, which rose in the annulus between the tubes and stirred the electrolyte. When initial attempts to anodically polish the entire inner surface of a long tube in one step showed that highly localized attack was obtained at the end electrically closest to the power source, the process was modified to yield two techniques. One using a translating shielded electrode was not developed beyond the test stage; the other using a non-translating unshielded electrode became part of the process.

The first technique consisted of electrically insulating the cathode from the solution except for several inches at one end. Removal of the W - 25 Re from the inner surface was pronounced only in the immediate vicinity of the exposed portion of the cathode. Uniform removal of metal along the entire inner surface was obtained by slowly tranlating the cathode through the tubing at a constant rate. Throughout a "run" air was bubbled from the bottom of the cathode through the inside of the W-Re tube and also from a dispersion tube in the solution at the bottom of the container. During several short tests a nearly smooth surface was obtained over several inches; however, the required time was excessive; consequently a faster procedure was sought.

In the method adopted, the entire inside was smoothed with a non-translating cathode by using 2 to 2-1/2 liters of a more dilute aqueous solution of NaOH (5 to 7.5 g. NaOH/liter) and the same cathode used for the translating cathode process. The cathode contacted the solution along its full length but its shorting to the W-Re tube was prevented by a braided glass sleeve and a spiral wrapping of small-diameter plastic cord, whose consecutive turns were separated by about 2 inches. During a "run" the cathode was continuously rotated at a speed of one revolution in 10 minutes to prevent masking of the surface by the plastic cord. As nearly as could be determined without destroying the tubing, this technique removed the metal uniformly along the surface (somewhat better results were obtained with the 7.5 g/liter solution). Large amounts (up to two- or three-mil thick surface layers estimated from weight losses) were frequently removed from as-extruded tubing before the desired smoothness was approached. Because of end effects and the inability to accurately measure ID and wall changes deep within the tube, only a qualitative indication of metal thicknesses removed can be given. Generally the removals were monitored by weight measurements which (assuming uniform metal removal) indicated whether reduction of wall was light (1 to 2 mils) or heavy (in excess of 4 mils). Since the amount of metal removed before noticeable smoothing occurred was a relatively high percentage (10-15 percent) of the initial wall thickness, the inner surface was only partially smoothed in the procedure adopted.

Only about 1/2 to 1-1/2 mil thick layers were removed so as to leave walls thick enough to be ironed substantially.

Every "run" began at or near room temperature; no effort was made to control the temperature during the bulk of the runs. In a few of the early runs temperature was maintained in the range of 10-30°C without noticeable benefits.

Honing

Although not incorporated into the process, honing of the interior surface was considered a possible means of reducing the striations on the internal surfaces. The small diameter of these tubes made honing and any other type of internal grinding or machining operation very difficult or impossible. Nevertheless, honing of a 9-inch length of Tube 218 by Honematic Machine Corp., Worcester, Mass., demonstrated the ability of this operation to smooth the internal surface of the tube. The internal striations were visibly reduced with a small loss of metal (2-1/2 w/o or 0.6 mils). The operation appears applicable to longer lengths. No further honing work was done in this program because of the projected cost of the operation and the apparent success of the process without it.

2. Swage Pointing

Swage pointing of the 3/8- to 1/4-inch W - 25 Re was performed at 1400°C as described on p. 41 of NMI-1264, using chopper dies. TZM inserts were again used when bare tubing was pointed for sinking or hard-mandrel drawing while the extrusion fillers were left in place for deformable-mandrel drawing.

Swaging of tubing with diameters below about 0.1-inch became difficult because of the problems of maintaining adequate heat in the tubing and adjusting the swager to prevent formation of flat fins. Better points were made in this size range using tapered approach dies rather than chopper dies. The tubing was still heated to 1400°C before swaging. Further work is necessary to develop a reliable swaging technique for small diameter tubing.

3. Measurements

Outside diameters were measured directly with micrometer calipers before and after drawing. Maximum and minimum readings were obtained at the front, middle and rear of the tubes.

Inside diameters were measured at the rear of the tubes using ball gages in combination with micrometer calipers. Although maximum and minimum readings were obtained, the maximum was considered reliable only when the tube was no more than a few mils out of round.

Maximum and minimum wall thickness were also measured at the rear end of each tube during the draw schedule using micrometer calipers with a cylindrical anvil. Such direct wall measurements are generally accurate within 1 mil providing burrs are removed from the tube ends. They, however, indicate the dimension at the end, which may be thinner than for the major portion of the tube as a result of end effects in electropolishing or drawing.

Tubing completed in pilot production was measured at both ends after the ends were cropped to uniform wall thickness. Thus the measurements are representative of the entire tube and indicate the tolerances the process can achieve (see Table 11). The OD, ID and wall were held to $\pm$ 1 mil in the pilot production.

4. Lubrication

The graphite-sugar solution used since the start of the drawing work remained the standard lubricant. The lubricating techniques used were those described on p.45 of NMI-1264. As was mentioned elsewhere (see Annealing, p. 25) the lubricant can leave a residue, which is difficult to remove, especially from the inside. Hence lubrication of the interiors of tubes drawn by the moving-mandrel technique was eliminated from the final process used for the pilot production of 1/4-20 tubing. The cleaning of the surfaces after drawing is discussed below (p.26).

5. Removal of Hard Mandrel

a. General

Moving-mandrel drawing, unlike stationary-mandrel drawing, produces a tube which is generally so tightly seated on a mandrel that it cannot be pulled off. Drawing conditions sometimes produced ironed tubes in which the mandrel was loose enough to be easily removed (Test 97, NMI-1264). However, such occurrences are rare and a supplementary operation is generally required to loosen the mandrel so that it can be removed. Light swaging, rolling and reeling rely on springback and/or wall thinning and are normally used to expand the tube away from the mandrel. One attempt at very light swaging at 480°C (after Test 183C), with conventional rather than chopper dies, was unsuccessful in this program: the material cracked and the M-2 steel mandrel (R_c 46) deformed. Rolling was not attempted. Reeling, which is especially useful for conventional metals, was used with much success for tungsten - 25 $^{w}/o$ rhenium in this program. In fact, the successful development of a reliable process to 1/4-20 tubing resulted from the development of a reliable reeling technique. Preliminary tests of ball swaging as a means of loosening the tube were also performed.

b. Reeling

The initial attempt at reeling W - 25 Re tubing from a mandrel, described in NMI-1264, p. 48 , was so successful that it appeared

to be a virtually trouble-free technique. Such facility seemed confirmed in Series 126 at the outset of this program. Subsequent attempts soon encountered problems such as cracking of the W - 25 Re (Test 133A), jamming of apparently loose mandrels (Test 139) and inadequate diameter increases. Some of the problems were attributed to defects in the reeling machine, which were eliminated by replacing worn parts and changing the drive mechanism. Incipient cracks and residual stresses introduced by the drawing step were considered responsible for fractures during reeling. Prevention of cracks is discussed elsewhere (see Annealing, Surface Preparation and Section E 4, p. 42.)

Improvement of the reeling step to the stage where removal of long mandrels was routine resulted only after the study of warm reeling, which is discussed in Section E, p. 36. This study established a combination of drawing conditions, reeling temperature, roll angle, roll spacing and mandrel material that permitted routine mandrel removal in the pilot production of 1/4-20 tubing from stock with OD about 0.30-inch and wall thickness about 25-30 mils. The success of the procedure depended on the use of 480°C for a reeling temperature and M-2 or M-3 steel mandrels with room temperature hardnesses ranging from R_c 60-65. A draw temperature of 450°C was used; however, temperatures to 510°C might also have been suitable. The rolls, which were not pre-heated (as in early tests), were coated generously with "Oildag". It is not known whether the Oildag influenced the technique but it was applied to prevent binding of tubes, which was occasionally observed in early reeling work.

The roll spacing was first established by trial, beginning with spacing a few mils smaller than that which would freely pass the tube. The gap was reduced a few mils at a time until several passes at a setting would loosen the tube. Once determined, the roll spacing for a certain size tube could be relocated and reused without additional trial passes. Roll angles were set so that the included angle formed by the axes was 134°.

In current practice the tube on the mandrel is placed in a horizontal furnace aligned with the guide bars on the reeling machine. The roll spacing is set as determined in the previously mentioned trial. When a thermocouple beside the tube shows the desired temperature, a reeling pass is made. In this manner, six to eight passes are performed at the same roll setting. The tube is then ready for removal from the mandrel. This combination of mandrel, temperature and machine setting did not produce diametral clearances greater than 2 mils in the tubing generated at the various stages of the schedule used to make the 1/4-20 tubes. However, these clearances were adequate to permit removal of the mandrels from three-foot lengths of tubing by pulling on the draw bench.

b. Ball-Swaging

Before the reeling was successfully established, ball- or planetary-swaging appeared to offer a means for providing the stresses necessary to thin the tube wall and open the tube. Basically, in the ball-swaging operation the tube wall is deformed by a ring of balls rolling

around the outer surface of the tube as the tube is drawn past the balls.[3] The configuration of the balls, their holder and the tube can be considered analogous to a ball bearing, in which the tube forms the inner race. One of the benefits of this operation is its capability for lifting strong materials from a mandrel as the tube is pulled (on a draw bench) through the rotating ring of balls. Since the tube cannot move past the balls until deformation has occurred, the applied force can be increased as in conventional drawing until stresses high enough for deformation are obtained.

Two attempts were made to determine whether ball-swaging would work as a means of loosening W - 25 Re ironed onto a hard steel mandrel. Material (torch heated to about 650°C before being fed into the machine) was successfully loosened after Draw Tests 164C and 164E. The clearance generated in a single pass was about the same as that obtained following multiple reeling. In particular, it was noted that the material appeared to be unusually deformable at the temperature used. (Prior experience of the operators had been with high-strength materials at room temperature). In addition, length increases and wall thinning showed that substantial ironing had occurred. Thus the method provides an alternative route to drawing. Possible use of the ball-swager for ironing was briefly explored in tests of four-inch lengths of Tube 226 over M-3 mandrels at 480 and 550°C (a resistance furnace was used for heating). The 480°C test was accompanied by a reduction of 4 percent, but many faint splits were observed. The 550°C test successfully gave a reduction of about 6 percent with improvement in wall uniformity.

6. Annealing

a. Stress-Relief Annealing

The basic annealing procedure to 1450°C using a Globar furnace was described in NMI-1264. At the beginning of the present program the need for stress-relieving the W - 25 Re tubing was not apparent. As the program evolved, recurrent cracking during or after drawing (see p. 42), particularly after sinking draws, indicated that stress relief was necessary. The first stress-relief treatments performed at 1200°C were beneficial but not completely effective. Subsequent stress-relief anneals at progressively higher temperatures showed progressively increased effectiveness until by the end of the program an anneal at 1400°C for 1/2 hour was being used after every draw. This anneal, while suitable for the draw schedule used in pilot production (nominal 10% reduction per pass, 30% cumulative), was still not completely effective for material worked toward 1/4-20 from 3/8-40 stock (about 60% cumulative reduction).

The 1400°C anneal can lead to formation of carbides from the residue of the lubricant. Procedures used to avoid or mitigate this problem are discussed below (p. 26)

b. Recrystallization Annealing

Improved drawability was sought during this program in order to lower the range of applicability of plug drawing, reduce the stresses on the tools or prevent cracking of the tubing. For these reasons the effect of recrystallization on drawability was of interest. Knowledge of the drawability of recrystallized material may also be useful in determining draw schedules for imparting special properties to the finished tubing. On the basis of microstructural analysis of the effects of various anneals (see Section V.C.2.b.,p.51)an anneal at 1800°C for 15 minutes was selected for test (see pp 37 and 40).

Approximately 27 inches of Tube 198 was annealed at about 1800°C for 15 minutes to provide recrystallized material for Draw Tests 140 and 141. A self-resistance electrical heating technique was used. The tube was held vertically within a Vycor chamber by two water-cooled electrical clamps, one at each end. Direct current heated the tube to the desired temperature, which was monitored by a manually operated optical pyrometer. The Vycor chamber surrounding the tube contained flowing argon - 15 percent hydrogen. The pyrometer reading correction for reflection, absorption and emissivity was estimated to be about 70°C. During most of the 15-minute anneal the pyrometer indicated about 1730°C along all of the tube except near the ends. The current-controlling device lacked a fine adjustment and several short duration overshoots, highest about 1970°C (corrected) for about 30 seconds, ensued before a nearly steady state was attained. The duration of the entire treatment was 25 minutes and at nearly steady state 700 amperes at 17.5 volts was required. After cooling the tube was shiny and clean.

The tube was cut to provide approximately equal lengths for a plug draw (Test 140) and a moving-mandrel draw (Test 141). Specimens were also taken for ring compression tests and metallographic examination, which indicated that the desired recrystallization had been achieved (see Section V.C.2.b(3)p52) but with some grain coarsening (Figure 9A). In the draw tests failure of the points indicated weakening of the tubing. Thus, this anneal was not successful but the possibility remains that drawability can be improved by a more effective anneal, presumably at lower temperature.

7. Postdraw Cleaning of Surface

The residue from the graphitic lubricant can lead to formation of hard carbides* during the stress relief at 1400°C. Means were sought for removal of the residue before the anneal or of the carbide after the anneal. Apparently the formation of the carbides can be avoided by having water present in the hydrogen atmosphere of the anneal.

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*Suspected areas were identified as carbides of tungsten by X-ray diffraction.

In one attempt to decarburize the surface, a drawn W-Re tube still coated with lubricant residue was annealed at 1400°C using wet (bubbled through water) rather than the usual dry (Dry Ice trapped) A - 15 H_2. The outer surface of the tube was clean after the anneal. Removal of the residue by vapor blasting prior to the anneal effectively prevented carbide formation on the exterior of tubes drawn in Tests 171C, D and E, 172D, E and F. Since no contamination was apparent on the outer surfaces of these tubes, vapor blasting and wet hydrogen annealing were made part of the process. The condition of the inner surfaces was not determined, but it is hoped that the wet A - 15 H_2 prevents contamination there also.

Prevention of carbide formation on the tube interior is still a problem following plug draws since the lubricant cannot be removed prior to the anneal without jeopardizing the integrity of the tubing (in a caustic soak - see below) or sacrificing the pointed material. Moving-mandrel draws can be performed without lubrication on the inside. Accordingly, moving-mandrel draws after Series 171 were made without lubrication on the inside and annealing was performed with wet A - 15 H_2. When some of the tubing finished early in the pilot production showed suspicious areas on the inner surface (viewed from the ends) aqua regia treatment of the interiors before annealing was incorporated into the process.

Fused Caustic Soak

Removal of the graphitic lubricant can be accomplished by soaking the drawn tube at 425°C in fused sodium hydroxide containing a trace of sodium nitrate. This treatment caused cracking of several tubes being cleaned of lubricant prior to anneals. In addition, the caustic does not rapidly attack the carbides formed in the 1400°C stress-relieving anneal. Although not applicable before anneals or useful against carbides, the treatment did nevertheless provide a means of finally cleaning the tubing after the drawing and annealing were finished. Consequently it was incorporated into the process used for the pilot production of 1/4-20 tubing.

D. Processes Investigated

1. General

Processes were developed for producing both 1/4-20 and 80-8 tubing. These processes were developed after consideration of alternative routes provided by several combinations of starting sizes and applicable drawing procedures. The route requiring the least drawing reduction is to extrude to the proper wall thickness with a slightly larger diameter, which can be reduced by a sinking or deformable-mandrel drawing step. This route, because it provides poor control of the ID, was not suitable for the 1/4-20 tubing where ID control was considered critical, but was suitable for the 80-8 tubing where ID control was considered less critical. In fact, because of the state of the W - 25 Re drawing experience at the beginning of this year's work, the sinking or deformable-mandrel routes were judged to be the

quickest, and most certain routes to the 80-8 tubing. Control of ID, wall thickness and surface finish required for the 1/4-20 tubing could be obtained only by the routes that incorporated draws in which ironing (wall thinning) occurred on a hard moving or stationary (plug) mandrel. Schedules incorporating these techniques were therefore planned and extrusions made to provide tubing which could be drawn by these schedules to 1/4-20 tubing having various reductions. One schedule was a continuation of a schedule established in last year's program for reducing tubing of the 3/8-40 size. Other schedules began with tubing of intermediate sizes between 3/8-20 and 1/4-20, as will be discussed below.

Sinking, although by itself considered inadequate for the 1/4-20 tubing, was frequently valuable in helping to reduce diameters so that eventually the tubing of various sizes could fit into a common tool schedule. Sinking was also used to obtain 1/4-inch outside diameter tubing following ironing passes which produced 20-mil wall tubing of larger diameters. A very light sinking pass after the final moving-mandrel draw corrected OD changes resulting from the final reeling step.

In addition to the above two sizes, tubing for re-extrusion or for applications elsewhere was sized primarily by sinking, by deformable mandrel drawing or by plug drawing, followed by sinking. The last route frequently followed schedules similar to those used for converting the 3/8-40 tubing to 1/4-20 tubing.

Problems in the various routes were solved or mitigated when encountered. Most notably, development of a reliable reeling technique eliminated the serious problem of removal of the mandrel after moving-mandrel draws and paved the way for the pilot production of 1/4-20 tubing (see Section IV.F, Epilogue).

Although a process was demonstrated for making nominally 80-8 tubing, pilot production of this size was prevented by lack of suitable quantities of satisfactory extruded material.

Many process steps used were common to nearly every piece of tubing drawn in this program. The material was pointed by hot swaging. The tubing was electropolished in a sodium hydroxide solution to remove about 0.5 to 1 mil from the outer surface. Its diameters, wall thickness and length were measured and the tube was visually checked for flaws. The surface was lightly oxidized to a dark grey, the lubricant was applied and dried; then the tube (with mandrel, if used) was placed in the preheated draw bench furnace with the point protruding through the opening of the preheated die. The grips were attached to the point (Figure 2) and about 5 minutes after a thermocouple beside the specimen had indicated the desired temperature (usually 450 to 510^{o}C), the tubing was drawn through the die. The drawn tubing was cooled to room temperature in air. The cool drawn tubing was then cleaned and its dimensions were measured and the tube was inspected. Partway through the program, the necessity for stress-relieving the W - 25 Re tubing during the drawing schedule became apparent, and intermediate anneals were used.

The various routes are discussed individually below. Table 5 is an index of the draw tests according to the drawing techniques. Some of the details regarding draw series are given in Tables 6 to 9; each table is devoted to tubing in a different size range. The detailed data regarding all the draw tests (including dimensions at different stages) are not reproduced in this report, but are available in two forms: the respective monthly reports, and a master compilation report on file at Nuclear Metals.

2. Routes to 1/4-20 Tubing

Three routes were successfully used to produce sound 1/4-20 tubing with good dimensional tolerances:

(a) Start with extruded tubing with OD near 0.3-inch and with wall thickness nominally 30 mils; draw by the moving-mandrel technique. (Total reduction about 30 percent).

(b) Start with material with a nominal 3/8-inch OD and with a wall thickness nominally 30 to 40 mils; sink and iron (either by plug or moving mandrel) to about 0.3-inch with about a 25-mil wall [now resembles stock used for (a)], then complete by moving-mandrel drawing as in the (a) route. (Total reduction about 50 percent).

(c) Start with material with a nominal 3/8-inch OD and with a wall thickness nominally 30 mils; plug or moving-mandrel draw to a wall thickness of 20 mils, then sink to a 0.250-inch diameter. (Total reduction about 40 percent).

The route outlined in (a) was developed into the process used for pilot production. Routes (b) and (c) were completed for only a few tubes and were not developed to the same high degree of reliability as (a).

A fourth route, plug drawing directly to the 1/4-20 size, was stymied by ductile failures of the drawn material (e.g. Tests 98F, 102G, 161E) when the OD and wall thickness became less than about 0.31-inch and 30 mils respectively (see p. 41). Failure was occurring at tensile stresses of about 200,000 psi, which is close to the reported strength* of the alloy.

a. Drawing to 1/4-20 Tubing from Nominal 0.3-inch OD x 30-mil Wall Tubing (Table 6)

Tubing initially having a nominal 0.3-inch OD with walls ranging from 24 to 30 mils in thickness was reduced to the 1/4-20 size using the moving hard mandrel technique (NMI-1264, p. 60) at temperatures in the range 300 to 510°C. Sinking steps were used where necessary to accommodate tubing to an ironing schedule. The following table gives the numbers of the draw tests leading to the process used for the Pilot Production from 0.3-inch diameter stock.

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* Chase Brass and Copper Co., "Rhenium-75 w/o Tungsten Data Sheet", April 1962. General Electric Co., Temporary Product Data Sheet, "GE-125 Tungsten-25% Rhenium Alloy", August 15, 1964.

Outcome	Wall Thickness of Starting Stock	
	Draw Test	
	24 mil	30 mil
Completed to 1/4-20; used for reeling tests; same size mandrel reused for consecutive draws.		167, 168 169,
Established Pilot Production schedule to 1/4-20.	134, 139	185 184
Showed capability for drawing below 20-mil wall thickness.	132 171, 172	
Not completed; mandrel deformed by reeling at 675°C.		186

The process sequence that finally evolved is given in Appendix I.

A few tubes, because of wall thinning during surface preparation, were too thin to provide much reduction in drawing to 1/4-20. These were drawn to even thinner walls (nominally 15 mils) in Series 132, 171 and 172, to demonstrate that the 20-mil wall was well within the capabilities of the procedure. Series 132 showed that tubing of about the 1/4-20 size could be sunk about 60 mils and very lightly ironed, but could not subsequently be heavily ironed at 300°C, even when the heavy ironing step was immediately preceded by a 1300°C anneal for 1/2 hour.

Problems revealed by the series relying on moving-mandrel drawing were as follows: tensile failure at point during attempted heavy ironing reduction at 300°C (Test 132E); spiral and transverse fracture during attempted heavy ironing reduction at 300°C (Test 134A); progressive microcrack formation (Series 134 and 139); longitudinal fracture induced by reeling (Series 133 and 184) or internal stresses (Series 144, 145 and 146); mandrel deformation (Test 169D and Series 182, 183, 186, and 187); molybdenum contamination (Series 139); carbide formation (series annealed at or above 1300°C with graphite lubricant). These problems are discussed in Section E.

b. <u>Drawing to 1/4-20 Tubing from Nominal 3/8-inch OD x 30- to 40-mil Wall Tubing by Gradual Reduction of Wall Thickness</u>

A complete route to the 1/4-20 size from 3/8-40 tubing was successfully demonstrated by Series 126, which gradually thinned the wall, beginning with one plug draw and thereafter using the moving-mandrel

and sinking techniques at 580°C. Gradual wall thinning was obtained nearly to the 1/4-20 size in Series 151 and 152, both of which relied more heavily on plug drawing performed at 510°C. When the cross section of the tube became so small that the forces necessary for plug drawing could cause tensile failure, ironing was obtained using moving mandrels at 450°C. Series 158, although discontinued after being processed only to 0.303 diameter with a 26-mil wall at 510°C, indicated that tubing extruded to a 3/8-inch OD with a 30-mil wall could be sunk heavily at first until it could enter the ironing schedule used for most other tubes initially of 3/8-inch diameter.

Series 102 was continued from last year's program (NMI-1264, Table 31) and encountered ductile tensile failure during an attempted plug draw (102G) to about a 20% reduction when the diameter and wall were less than 0.31 x 30. Material drawn in 102G suffered point failure during a subsequent attempt at a heavy reduction (20%) in 102H.

c. <u>Drawing to 1/4-20 from 3/8-30 by Reducing Wall Early in Series; Remainder of Reduction by Sinking (with Plug)</u>

Plug drawing, desirable because it avoids the mandrel removal problem, is plagued in the smaller diameter thin-wall tubes by tensile failure of the drawn tubing. Plug drawing to 20-mil wall in diameters larger than 0.31 -inch was considered possible in view of the results of Test Series 70 (NMI-1264, Table 23). Tests 191A and 192B were initial probes to determine the feasibility of making 1/4-20 tubing by first plug drawing to 20-mil wall in larger diameters and subsequently using sinking or <u>very light</u> ironing passes to achieve final size. Any ironing in these final passes may have been caused by the mandrel used to provide insurance that the correct inside diameter would be maintained. The clearance between the die and the mandrel was equal to the thickness of the wall of the tubing being sunk; consequently, whatever ironing occurred was minimal.

Test 191A, an attempt to go from a 28- to a 20-mil wall in one pass (attempted reduction about 32 percent) was terminated by tensile failure of the drawn tubing. Tests 192A and B successfully produced 20-mil wall tubing after a 32 percent cumulative reduction. Test 193A, which began as an attempt to duplicate Test 192A, required forces so high that the tube necked slightly and would probably have fractured if the mandrel had not broken loose from the draw bench. The necking in Test 193A showed that even the 10.5 percent reduction of Test 192A had required draw forces perilously close to the tensile strength of the material. Thus, it seems doubtful that the plug drawing schedules used in Series 192 and 193 will consistently produce 20-mil wall tubing. Modification of the schedule to exploit strengthening of the material by drawing might, however, permit reliable plug drawing to a 20-mil wall. The possibility that the material might be strengthened enough to permit its plug drawing in this size range was suggested by Test 193C. This plug draw pass produced a 14.6 percent reduction although the cross section was less than when the tensile necking occurred during an attempt at a lower reduction of the same tube in the as-extruded condition

(see 193A above). Exploitation of this work-strengthening was explored in Series 194 which suggested that less ambitious reductions in early plug draws might strengthen the tubing enough to permit heavier reductions of subsequent plug draws. Such an approach might improve the reliability of the technique to the extent that the 20-mil wall could, as in Series 192, be obtained by plug drawing. If plug drawing cannot be applied, the desired sequence involving ironing to 20-mil wall before sinking to final diameter might be accomplished by incorporating the moving-mandrel technique. The reeling operation is somewhat easier for diameters near 3/8-inch than for diameters near 1/4-inch.

d. Problems with Alternative Routes

Problems encountered during tests on the alternative routes, relying on plug drawing, are summarized as follows: tensile failure of the drawn tubing (102G, 135A, 161E, 191A, 193A); plug deformation (126A, 128B, C, 135C, 138B); longitudinal cracking (any length) in reeling (Series 126, 128, 135, 138, 151, 159, 160, 162), in sinking (149G), or during moving-mandrel draws (128G, 138D); moving mandrel deformation (160C, 194G); failed linkage of plug to draw bench (128D, 135A, B); lubricant failure (152D). These problems are discussed in Section E.

3. Routes to 80-8 Tubing (Table 8)

a. Techniques

Sinking and deformable-mandrel drawing were the techniques selected for making the 80-8 tubing from stock extruded slightly oversize. During the program the main routes tested were as follows:

(a) Sink from about 85 mils diameter (Series 129 and 131) or from about 130-mil diameter (Series 124 and 125).

(b) Deformable-mandrel draw from about 130 mils diameter with solid or hollow mandrel (Series 123 and 148).

(c) Deformable-mandrel draw from about 100 mils to about 85 mils OD with molybdenum-CaC_2 composite filler, remove mandrel and sink (Series 153 and 166).

The procedures are the same as those described for larger sizes of W - 25 Re tubing. Sinking and deformable-mandrel drawing tubing slightly larger in diameter with a wall of the desired thickness were at first hampered by the difficulty in removing the solid inner extrusion filler (which also could serve as a deformable drawing mandrel) from long tubes. Larger tubing could be cleared more easily; therefore sinking from larger sizes was attractive until the advent of the easily-removed CaC_2 extrusion core. Such cores made practical sinking or deformable-mandrel drawing of tubing extruded nearly to final size.

b. Results

(1) Sinking from Nominal 0.1-inch OD x 10-mil Wall. The feasibility of sinking tubing with diameters in the range of 70 to 130 mils, which brackets the size desired, was demonstrated by Series 124, 125, 129 and 131.

Diameter changes of about 10 mils per pass were obtained for two passes in each of Series 124 and 125 which used stock* having initial diameters of about 135 mils. Draws in those series were performed at 580°C with intermediate anneals at 1200°C. Diameter changes of about 4 mils per pass accumulated to about 15 mils in Series 129 and 131 which used tubing** initially with about 83 mils outside diameter. The latter series used 1300°C stress reliefs after each pass and for each series the A and B passes were at 580°C, while the C and D passes were at 300°C.

Further sinking of tubing produced in the four series was prevented by recurrent point fracture.

(2) Deformable-Mandrel Drawing from Nominal 0.1-inch OD x 10-mil Wall

(a) Solid Molybdenum Mandrel. One length of W-Re tubing from Tube 171* was drawn at 580°C (Test 123) with the extrusion molybdenum still in place and acting as a deformable mandrel. The tubing drew nicely and produced material with a 95-mil OD from stock with a 132-mil OD.

After the last draw the molybdenum filler was removed from a 9-1/4 inch length by acid drilling.*** The cleared section was found to be crack-free and impervious to helium. This series shows that small diameter W-Re tubing can be sized on the OD without drastically reducing the wall uniformity.

After the last pass (123D) the material developed a tendency to crack longitudinally when being cut transversely. This cracking tendency was symptomatic of severe internal stresses and indicated the need for a stress-relieving anneal after draws of this type.

(b) Hollow Molybdenum Mandrel. Test Series 148 was made on a section of tubing from a secondary extrusion (Tube 216) ****

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* Tube 171 primary extrusion at 1600°C, 16:1 R; secondary extrusion at 1200°C, 9:1 R.

** Tube 182 primary extrusion at 1600°C, 16:1 R; secondary extrusion at 1200°C, 25:1 R.

*** It should be mentioned that the acid drilling of a solid core is a slow process (~3/8" of length per hour). The time for processing long lengths permits noticeable attack of the W - 25 Re.

**** Primary extrusion at 1600°C, 16:1 R; secondary extrusion at 1200°C, 9:1 R.

with a calcium carbide core within an inner molybdenum filler. The carbide was removed before drawing, which was then accomplished with the inner molybdenum acting as a deformable mandrel. After the second draw the axial hole formed by the removal of the carbide was closed. The drawing was continued until the OD had been reduced a total of 37 mils to a final size of 88 mils. Further drawing was not attempted since diameter changes no greater than 15 mils were anticipated for producing the 80-8 tubing.

(c) Deformable–Mandrel Drawing with CaC_2, Then Sinking. An important step in the process for making 80-mil diameter W - 25 Re tubing was demonstrated with Test Series 153 and 166, which produced lengths of tubing with about 80 mils OD from 1200°C and 1500°C re-extrusions. This tubing was obtained by drawing at 510°C re-extruded stock (Tubes 223* and 224**) that still contained the Mo-CaC_2 inner extrusion fillers. Total outside diameter changes of 12 to 14 mils were obtained by four passes in each series, with a 1400°C anneal after each pass. It is interesting to note that the wall of the tubing was thinned by these passes. Following the fourth deformable-mandrel pass the CaC_2 was removed by acid drilling (p. 10), then the molybdenum was rapidly dissolved by flowing HNO_3 - H_2SO_4 through the axial hole created by the removal of the CaC_2. Series 151 was then completed to a nominal 80-mil OD by a sinking pass. An attempt to finish Series 166 by a similar pass was prevented when the point was damaged while it was being swaged to pass through the sinking die. The remaining tubing was short and drawing was discontinued.

(d) Status of Processes for 80-8 Tubing. Sinking (hollow tubing) or deformable-mandrel drawing (CaC_2 core) from slightly oversize tubing are the routes that offer the most promise at this time. Diameter reductions of 5 to 7 percent per pass at temperatures ranging from 300 to 580°C appear satisfactory for obtaining 15 to 20 percent total diameter decreases when stress reliefs are provided after each pass. The routes using solid deformable molybdenum mandrels are impractical because of the time necessary to clear the ID after drawing. (The long time required would not be too objectionable except that the attack on the tubing by the acid used to clear the ID over extended periods noticeably affects the wall thickness.) If the CaC_2 is removed before completion of drawing, the remaining hollow molybdenum must be removed before it closes to the extent of no longer permitting ready attack by acid.

The problems foremost in application of the routes discussed above for making 80-8 tubing are removal of inner filler(s) and swaging of the points. Drawing should be straightforward if the re-extrusion process can provide

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* Primary extrusion at 1600°C, 16:1 R; secondary extrusion at 1200°C, 16:1 R.

** Primary extrusion at 1600°C, 16:1 R; secondary extrusion at 1500°C, 16:1 R.

tubing with a continuous CaC_2 core (p. 11). Swage pointing can presumably be improved by refinements in tool adjustments and in heating procedures.

Sinking of tubing ironed to 7- to 8-mil thick walls, in diameters as close as possible to 80 mils, is worth exploring as a back-up route in case ID control becomes more critical.

4. Drawing for Special Uses

a. Irradiation Stock (Table 9A)

Series 99 and 103, which began with plug draws of 3/8-40 material to about a 40% cumulative reduction, were completed to the desired size, 0.255-inch OD x 30-mil wall, by sinking passes which decreased the diameter about 60 mils. Draws in Series 99 and 103 were performed at 580°C and 510°C respectively and every sinking pass was preceded by a 1200°C, 1/2-hour anneal. These series, together with similar but less successful series (98, 100 and 101), utilized material extruded at 1600°C to a 16:1 reduction. Problems encountered in these draws included: tensile failure of the drawn tubing during plug draws (98F and 102G); mandrel deformation in plug draws (99F, 100F); cracking during sinking (98E, 100I and 101G); cracking during plug drawing (99E). These problems, which were encountered in other draw series, are discussed in Section E.

Other material extruded at 1600°C but to a 25:1 reduction (Tube 200) was ironed on a moving mandrel and sunk from about 0.31-inch OD x 33-mil wall to 0.255-inch by 30 mils in Series 147, which incorporated a 1380 - 1400°C anneal before each sinking step. Similar drawing of a companion series (146) was not completed because the tube cracked lengthwise 14 hours after pass "B" (before it could be annealed, p. 42). Additional irradiation stock sized to 0.255-inch OD x 30-mil wall was made by sinking material with 3/8-inch OD x 30-mil wall extruded at 1600°C using a 16:1 reduction (Series 156) or a 25:1 reduction (Series 154 and 155).

Another batch of irradiation stock was obtained by sinking (Series 179, 180 and 181) material initially 0.31-inch OD and 30-mil wall made by extrusion at 1600°C to a 16:1 reduction. Series 154, 155, 156, 179, 180 and 181 all incorporated 1400°C, 1/2-hour anneals after each pass and encountered no problems.

b. Re-Extrusion Stock (Table 9B)

The deformable-mandrel technique (NMI-1264, p. 60) was used extensively to size tubing to 0.375- to 0.380-inch OD for re-extrusion. The starting material was nominally 0.38- to 0.40-inch OD extruded at 1600°C to a 16:1 reduction. Usually one draw was sufficient (Series 136, 137, 157, 173, 175, 176 and 190), but two draws were used for Series 142. The tubing had been extruded at 1600°C at a 16:1 reduction.

Smaller diameter stock was also sized in the same way for re-extrusion. Series 143 produced a material with a 0.313-inch OD from extremely oval (OD range 0.308- to 0.340-inch) stock extruded at 1600°C at a 25:1 reduction. A smaller diameter tube extruded at 1600°C, 16:1 reduction, was drawn to 0.302-inch OD from about 0.31-inch OD in Series 177 and 178.

The above deformable-mandrel draws were made at 510°C with or without removing the graphite extrusion core and were followed by stress-relief anneals except for Series 142 and 143. Series 137 and 142 were made with a tube that had been severely bent after extrusion. Before drawing, the tube had been partially straightened by hand forging near the swaging temperature.

c. Miscellaneous Draws (Table 9C)

Two additional tests were made which concluded tests begun last year (NMI-1264). Test 70C was partially successful in plug drawing to an additional 6.6 percent reduction material already plug drawn to a 26 percent reduction, and showed that the M-3 steel R_c 60-65 was promising as a plug material (cf. p. 41) Test 77C unsuccessfully attempted to test another hard plug material by plug drawing to an additional 12 percent reduction material already drawn to a 3.8 percent reduction (NMI-1264, p.156) after extrusion at 1800°C.

E. Problems and Their Resolution

1. Mandrel Removal after Moving-Mandrel Drawing

Following the success with the moving-mandrel technique and reeling at 600 to 650°C in Series 126, much difficulty was frequently encountered in removing the mandrels after the drawing passes. Earlier tests (Tests 62 and 63, NMI-1264) had encountered mandrel bending, which makes mandrel removal difficult. During tests such as 146A, 147A and 147B, reeling was found to be inadequate. These tests were preliminary draws in series aimed at supplying material with a 0.255-inch OD and a 27- to 30-mil wall for irradiation tests. These three draws were made at 510°C using hard mandrels of an unidentified steel, the same as those used for draws in Series 126 below 0.25-inch inside diameter. Although some lifting of the tubes was apparent after reeling, mandrel withdrawal was very difficult. After Test 147B, the mandrel was found to have a 4-mil diameter variation along its length. (Apparently this mandrel was deformed by drawing and/or reeling.) Subsequent tests using M-2 steel (R_c 46-50) mandrels also encountered deformation problems. The solution of this problem, through the combination of mandrel material and working temperature, is discussed after a summary of an unsuccessful attempt to enhance drawability by a recrystallization anneal.

The recrystallization anneal of Tube 198 is described above (p. 26); the selection of the conditions and the evaluation of the annealed tube are covered below (p. 51). Portions of the annealed tube were used for moving-mandrel and plug draws in Series 141 and 140 respectively. An attempt at a 22 percent reduction for the initial moving-mandrel pass was abandoned because the point kept breaking (as in the companion attempt at plug drawing in Series 140, p. 40). The tube, although it drew satisfactorily through two passes (B and C) at lower reduction using a different type point,* was susceptible to intergranular fracture and cracked fully during reeling after pass C.

Since the 510°C draw temperature was in the range where most iron-base alloys soften rapidly, lower drawing temperatures were tested to determine whether they could be used. Portions of Tube 195 were drawn with the same tools at 510 and 300°C in attempts to simulate the 20 to 30 percent reduction contemplated for the pilot production of 1/4-20 tubing from slightly oversize extrusions. At 510°C (Test 133A) a 20 percent reduction gave sound tubing, which cracked lengthwise during the subsequent reeling at 600 to 650°C. There was some indication that unnecessarily excessive reeling was responsible for the crack. The attempt to achieve a similar reduction at 300°C (Test 134) was unsuccessful. Apparently the 50 percent increase in force resulting from the lower temperature was enough to break the tubing.

The lower temperature (300°C) was successfully used for light moving-mandrel draws of tubing in the 1/4-inch OD range having walls about 14 mils thick (Tests 132C and D). The success of these tests probably results from the use of very low reduction. Test 135A was completed as a moving-mandrel draw with a reduction near 10 percent at 300°C.

Drawing temperatures approaching 300°C are more agreeable to the tool materials and the drawing lubricants. Because of the fracture in Test 134, 300°C is not considered reliable.

On the basis of reeling experience in draw tests subsequent to Series 126 (e.g., Series 139), mandrel deformation was suspected to be the cause of much of the difficulty in mandrel removal. Therefore, the problem narrowed down to finding a mandrel material that would not permanently deform at temperatures where drawing and reeling could perform their respective tasks. Mandrel deformation was subsequently eliminated through use of M-2 or M-3 steels hardened to R_c 60 to 65 and moderate reeling temperature (480°C). The tests of various mandrel materials and reeling conditions that established a procedure to assure mandrel removal are summarized in Table 10.

Since the best mandrel material at the start of the study was M-3 steel (R_c 60-65), the initial tests were concerned with learning what could be accomplished with it. The first set of reeling tests was performed by torch

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* The point was the swage-formed internal shoulder which engaged a shoulder on the moving mandrel which itself was gripped and pulled through the die (see Figure 16A and p. 43 of NMI-1264).

heating the tubes on the mandrels to various temperatures which were measured by temperature-indicating crayons. Higher temperatures for easier working were favored at first; even though the mandrels might soften, it was hoped that they would remain hard long enough for the W-Re to be worked. As soon as the crayons indicated attainment of temperature within about 50°C of that desired, the tube on the mandrel was reeled. Although the rolls had been heated in earlier reeling, roll heating was abandoned in recent work because heat transmitted from the rolls was affecting the roll bearing lubricant. Moreover, these tests at dull red heat showed that the tube lost little heat in its passage through the machine.

During these tests, mandrel removal by reeling was accomplished after both light drawing reductions (167B, C, 168B, C and 169B, C) and a heavier reduction (169D). All these draws were performed with the same mandrel of M-3 steel, with an initial hardness of R_c 65. These successes in reeling led to the following conclusions:

(1) Lifting does not occur at 260°C.

(2) Lifting can occur at 500 to 680°C.

(3) The tube temperature remains fairly constant during a reeling pass.

(4) Three passes at the same roll setting can lift the tube from the mandrel enough for manual removal.

(5) The M-3 steel softened to R_c 35 and decreased about 3 mils in diameter after repeated use at the higher temperatures.

(6) Changing the roll angle to produce a more closely spaced track (like machining a finer thread) may have made the operation more effective. Rolls were reset so axes formed included angle of 144° rather than 134°.

Further testing of M-3 (R_c 60) mandrels used a resistance-heated furnace and a thermocouple for better control of temperature prior to reeling. These tests, which followed draws 172B and 184A, showed that mandrels could be removed readily after reeling at 480-510°C following 450°C draws. The lower draw temperature was used in an attempt to enhance springback.

Using similar techniques, mandrels of other materials were tested at different temperatures. Reeling at 425°C could loosen H-12 steel (R-50) mandrels without affecting their hardness or dimensions following 425°C draw

tests 171E and 172F which merely sank or very lightly ironed the tubes onto the mandrels. The H-12 steel softened and deformed, however, when reeled at 480°C.

M-2 steel mandrels with hardness of about R_c 48 appeared satisfactory for reeling at 510°C (171B, 172C, 182C), but softened and deformed at 650°C (183C). Lescalloy 718 mandrels were deformed after reeling at 480°C (171C) although the hardness remains unchanged. This material was found to be too weak for use with the W - 25 Re.

The above tests established conditions that permitted routine mandrel removal following moving-mandrel draws used to make 1/4-20 tubing. In the Pilot Production program, M-2 or M-3 steels with room-temperature hardness of R_c 60-65 were successfully used for moving-mandrel drawing at 450°C followed by 6- to 8-pass reeling at 480°C.

In spite of the encouraging results in the above reeling tests and the Pilot Production, reeling is still marginal in the sense that the tube clearances generated were only about two mils and a slight bend could easily cause jamming. Moreover it was not always certain exactly when the tube was adequately lifted.

The difficulties with the reeling operation appear to be caused by the inability to work the W-Re adequately at a temperature low enough to prevent deformation of the mandrel. Presumably if a mandrel could be obtained that is non-deforming at a temperature high enough to permit adequate working of the W-Re under the loads obtainable in the reeling machine, reeling would be easier and long tubes could be pulled manually off the mandrels. Since, however, such mandrels have not been obtained and since they require a tough, moderately ductile material, superior in strength to the W-Re (and as yet unavailable, at least in a form suitable for mandrels), it does not seem likely that reeling at higher temperatures will be successfully accomplished at this time.

Greater deformation of the W - 25 Re might be obtained if the applied force could be increased; however, limitations in the reeling machine available for this program have prevented the test of such a route. Continued efforts should be made to optimize the technique for W - 25 Re by adjusting roll angles, roll spacing, temperature and mandrel condition until manual removal of long mandrels becomes routine.

Exploratory tests on the use of ball swaging to loosen a tube on a mandrel are discussed in a previous section (p. 24).

2. Plug Deformation

One of the major problems in warm plug drawing tungsten - 25 w/o rhenium has been deformation of the mandrel at the land of the die.

Plug drawing is preferable to moving-mandrel drawing because it eliminates the necessity for mandrel removal. Tubing with outside diameters larger than about 0.32-inch, and walls thicker than about 30 mils can be drawn to low reductions (about 10 percent) per pass with a reasonable chance of success at 510°C. However, in the size range where plug drawing is suitable, the M-2 steel mandrels (R_c 46-50) used since the start of the program frequently deformed (e.g. 99F and 100F). Various methods for avoiding plug deformation are discussed in the following subsections.

a. Lower Temperature

Efforts to prevent mandrel deformation by drawing at lower temperatures were not successful. Tests 128B and C were peformed at 450 and 425°C respectively. In the latter, in spite of the low reduction, the M-2 steel mandrel still necked about 2-1/2 mils. Test 128D, at 400°C, was started as a plug draw but after 4 inches had drawn, the draw forces rose so high that the mandrel fractured in the thinner, threaded portion where it was fastened to the rear of the draw bench. The mandrel used as a plug in Test 128D showed no pronounced necking; however, the duration of the plug-drawing portion of the test was too short to demonstrate that necking would not have occurred. The high forces required for Test 128D might eventually have caused fracture of the W - 25 Re, since they were still rising when the mandrel failed. As soon as the mandrel became free to move with the tube the forces dropped to about half those required for plug drawing. A heavier threaded section will be required on mandrels of this size if plug drawing at 400°C is to be tested further. (The threaded portion of the plug was originally designed for a mandrel with a shoulder bearing on the point (cf. Figure 16A, NMI-1264) for moving-mandrel drawing and hence was thinner than required for plug drawing.)

b. Recrystallization of Tubing

Plug deformation might also be avoided if the drawability of W - 25 Re itself could be enhanced. The ring compression tests suggested that recrystallization might accomplish this.

Test 140B was an attempt to plug draw a piece of tubing extruded by the reference process (1600°C, 16:1 R) and recrystallized by an 1800°C heat treatment described earlier (p. 26). The hoped-for improvement in drawability of recrystallized material did not materialize. The tubing appeared to be weaker than the as-extruded material (compare Test Series 138), as indicated by repetitive point fracture. The appearance of the fracture surfaces was suggestive of intergranular fracture.

c. Alternative Mandrel Materials

Two alternative mandrel materials, M-3 steel and a proprietary alloy (NM-100), both hardened to R_c 60-65, were tested. The

former was used at 510°C while the latter was used at both 510 and 580°C. In two of the tests (136A and 128A) mandrels made from the proprietary alloy deformed. The deformation, however, occurred where the mandrel end contained a center drill hole. Another test (127A) of the same mandrel material was terminated by fracture of the mandrel where it was attached to the rear of the draw bench. A fourth test (77C), although prematurely terminated (by tensile failure of the tubing) before significant drawing occurred, indicated that the proprietary alloy was capable of resisting deformation. The behavior of the M-3 steel in Test 70C was also very promising; however, as with the NM-100 the test was too short; moreover the reduction was rather light.

These materials merit further consideration. Since M-2 steel can be hardened to hot hardness values approaching those of M-3, it should be adequate if used in a harder condition than the R_c 46 of the M-2 plugs tested in this program. A harder working tip may be sufficient.

Plug deformation might be circumvented with conventional carbide plugs During one test last year (Test 41 , NMI-1264) a conventional tungsten carbide-tipped stationary mandrel failed where the carbide was brazed to the steel shank. It should be possible to use a braze more suitable for the drawing temperatures. This approach, although enticing, was abandoned when successes with the moving-mandrel technique made further development of plugs unnecessary for this program.

3. Tensile Failure During Plug Drawing

Ductile tensile failure of the drawn material was also observed during plug draws and became quite frequent when the tubing dimensions were below 0.31-inch OD x 30-mil wall (Tests 98F, 102G, 161E). Tubing from Series 98 and 102, previously drawn to cumulative reductions of 28 and 42 percent respectively, failed in attempted single-pass reductions of about 27 and 33 percent respectively. Such high reductions were attempted because of the sizes of tools on hand; they generated draw forces that exceeded the tensile strength of the tubing. Tests 98F and 102G were immediately preceded by a 1200°C, 1/2-hour anneal. Both tubes failed at about the same stress while attempting the same reduction. Test 161E, an attempt at a reduction slightly below 10 percent, failed at about 200,000 psi stress, which is reasonably close to the tensile strength of the W - 25 Re (see p. 29).

Recrystallized material, rather than being more easily drawable and consequently subjected to forces more safely below its tensile strength, was even more susceptible to failure in Test 140B than when drawn in the as-extruded condition (Series 138). The anneal was performed at 1800°C as described on p. 26 . The appearance of the surfaces resulting from the drawing fracture suggested intergranular fracture had occurred.

4. Crack Formation

Longitudinal cracks, short or full-length, became a serious problem early in this year's program, beginning with draws which continued the series begun last year (100 and 103). As the program progressed, longitudinal cracks were encountered during various types of drawing, during reeling and during fused caustic treatment or other handling after drawing.

Internal stresses combined with numerous stress riser sites provided by the surface striations are considered responsible for most of the cracks. Incipient cracks possibly formed by such stress concentration are shown in Figure 3. Such stresses and incipient cracks may cause major cracks during reeling if proper conditions are not maintained. Sinking produced material with large internal stresses whose effect was most dramatically apparent after Test 146B when the tube developed a banana-peel fracture 14 hours after sinking while awaiting a stress relief. Internal stresses created by sinking and aggravated by thermal shock were responsible for cracking observed during immersion of tubes in fused NaOH (for lube removal) following passes 144B and 145B. Many of the cracks in drawing occurred when large diameter changes took place during attempts at heavy single-pass ironing reductions (e.g. Test 132E, 133A, 165C) or during sinks after large cumulative ironing reductions (e.g. Test 98F, 100I, 101G, 149G). Large cumulative ironing reductions also eventually yielded cracked material (Test 135E) possibly as a result of improper reeling conditions. Reeling imposes harsh conditions on the tubing and should be minimized. During Test 135E and other tests where cracking occurred as a result of reeling (e.g. 168E and F, 169F) the reeling technique still required improvement. Such improvement appeared to have been achieved by the process used for Pilot Production. Complete separation of the various interrelated factors influencing cracking was not within the scope of this program, but precautionary measures were established which prevented cracking during the Pilot Production.

Surface finish improvement and stress-relief anneals were the two approaches explored for reducing the cracking frequency.

Smoother surfaces were expected to reduce the sites for stress concentrations. Although surface smoothing by itself appeared unable to prevent cracking, it was incorporated into the process and was considered desirable for other reasons (prevention of lube entrapment and surface improvement).

Annealing gave the most promise of preventing cracking and was explored early in the program, beginning with a 1200°C anneal which enabled reductions of over 50 percent in Series 99 (at 580°C) and 103 (at 510°C). Series 99 was a companion to Series 98 (at 510°C), which used no anneals and ended with a cracked tube. It should be pointed out that the stress-relief anneals were used only after about a 40 percent reduction was achieved in each series by plug drawing. All subsequent passes were sinks and were preceded by 1200°C, 1/2-hour anneals.

Less frequent anneals at 1325^{o}C were not completely reliable (Series 138, 144, 145, 146). Even 1400^{o}C does not appear to guarantee that cracking will not occur (Series 165). The use of 1400^{o}C, 1/2-hour anneals after every drawing pass greatly benefited the process, as shown by the successful sinking reductions of over 0.1-inch in Series 154, 155 and 156 and the absence of cracking during the Pilot Production draws. It appears quite reliable to cumulative reductions of about 30 percent (except for 162). When reductions of about 50 percent were sought using the 1400^{o}C anneal after every pass, occasional cracking was again observed (Series 151, 159 and 194). These three series had unusual backgrounds: the tubing had been worked down from 3/8-40 stock extruded at 1600^{o}C to reduction of 25:1 for Series 159 versus 16:1 reduction for Series 151 and 194. Large sinking reductions (60 to 70 mils) had been used before ironing on plugs occurred. Fractures occurred during reeling after moving-mandrel draws despite the use of a 1400^{o}C, 1/2-hour anneal after every draw. These tubes had been centerless polished on the outside, pickled by H_2O_2 on the inside, and electropolished on the inside and outside. The cracking in these tubes may indicate that the 1400^{o}C, 1/2-hour anneal is not adequate for heavily worked tubes.

Since the evidence suggests that the 1400^{o}C, 1/2-hour anneal may not provide adequate stress-relief at high reductions, longer anneals should be studied. Temperatures higher than 1400^{o}C should be approached with an awareness that recrystallization can occur and may be detrimental.

Surface smoothing and 1400^{o}C, 1/2-hour annealing have greatly improved the reliability of the process for drawing W - 25 Re tubing particularly in the process used for pilot production. These steps are considered essential for the production of good tubing from extruded stock and should not be omitted from the process.

F. Epilogue - Pilot Production of 1/4-20 Tubing

The development program culminated in the pilot production of 51 feet of 1/4-20 tubing, including lengths near 3 feet, under a purchase order from Pratt and Whitney's CANEL Operation. Almost all of this tubing was drawn from stock extruded to 0.29-inch OD x 30-mil wall. The process is discussed on p.29 and the process sequence given in Appendix I.*

The drawing histories of 51 feet of tubing in the pilot production are given in Table 11. It is seen that some details of the process (notably the postdraw anneals) had not quite been standardized for some of the earlier tubes, which thus helped establish the process; all the subsequent tubes (starting with D196) demonstrated a reproducible process. These tubes were all found to be sound by the following non-destructive tests: helium leak test, fluorescent dye penetrant, radiography and eddy current. Some tubes also tested by ultrasonic means were found to be sound. Occasional short-crack indications found near the tube extremities were attributed to cutting

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* Subsequently 13 feet of short lengths with the same final diameters was prepared by somewhat different processes. Some of the 13 feet completed series initiated in the development program and required 50-60% reductions. A portion of this tubing received a recrystallization anneal before the last ironing pass at 1500 or 1550^{o}C.

or handling rather than to drawing. Such defects are considered preventable. Some of the radiography indicated spotty wall thinning (mottling on the radiograph film): the extent of such thinning was not determined and is considered to be less than 1 mil.

The only losses in the drawing by the standardized process were in the few inches (about 3) consumed in the point of every length and the occasional fraction of an inch that had to be cropped from the rear end because of a crack; all of the lengths survived the drawing operations without failure. Thus, the over-all loss in this working is estimated at about 10 percent.

These results for the pilot production drawing can be combined with other data from this program to indicate an over-all yield of about 70 percent of sound tubing from the sintered sleeve. In the primary extrusion, the end defects are responsible for a loss of about 10 percent when an 8-inch long sleeve is used (since the absolute quantity of end defects seems to be independent of sleeve length, the fractional loss is doubled for a 4-inch sleeve). Allowance also has to be made for an additional loss of 10 percent in the conditioning of the extruded surfaces by abrasive polishing and electropolishing. With the likely exception of the polishing losses, the various losses (extrusion defects, drawing points, cropping) can be processed for rhenium recovery.

V. EVALUATIONS

A. Introduction

Evaluations were directed towards characterization of the sintered sleeves and fabricated tubing, as a continuing guide for the fabrication program. These evaluations included determination of the following properties: hardness, density and microstructure of extrusion sleeves; ring compression ductility and annealing behavior of extruded tubing; and annealing behavior of drawn tubing.

B. Experimental

The sampling and testing procedures developed earlier were used throughout the current program. These procedures were described in NMI-1264 and, hence, are reviewed here only briefly.

1. Sampling

a. Extrusion Sleeves

Ring samples of the W-Re extrusion sleeves were supplied by the vendors. These rings were cut at a convenient stage in the processing (i.e. before or after sintering); in either case the ring was always sintered with the parent sleeve and had the same processing history. About one-quarter of the ring served for hardness measurements and metallography, the remainder for measurement of sleeve density.

b. Extruded Tubing

Pickling of the outer molybdenum filler permitted identification of the uniform section of each tube. The extrusion end defects were then cropped. Ring samples 0.2-inch long were obtained from the front and rear of the cropped tube. Up to fifteen samples were cut from each location, the number depending on the evaluations to be performed.

c. Drawn Tubing

Drawn tubing was sampled in the rear portion. As in the case of extruded tubing, the number of rings cut from the drawn tubes was determined by the evaluations to be performed.

2. Ring Compression Tests

The 0.2-inch long compression test specimens were first deburred and then electropolished in 2% NaOH; the electropolish removed approximately 1 mil per side. This method was found in earlier work to

give higher apparent ductility and to reduce scatter in the results (see NMI-2121, p. 7 and Table A-IV).

Figure 4 is a schematic diagram of the apparatus used for the ring compression tests. The specimens were placed on the bottom anvil in the position shown in the figure, heated to the test temperature and equilibrated 5 minutes. The specimens were then compressed on a transverse axis at 0.1-inch per minute punch travel until failure or until the applied load reached 600 pounds. In order to prevent surface contamination during test, the specimens were coated with a colloidal suspension of graphite in alcohol ("alcohol-Dag") and the furnace flushed continuously with tank-purity argon. The deflection at failure and the load at 0.025-inch deflection were read from load-punch travel curves recorded during the test.

Deflections measured in the ring compression tests were normalized on the basis of the fraction of maximum possible deflection. This fraction is defined as the deflection divided by the maximum possible deflection. For ring specimens the maximum deflection is equal to the ID, or the OD minus twice the wall thickness. Loads at 0.025-inch deflection were normalized to unit length.

3. Hardness

Specimens for the hardness measurements were mounted in Bakelite, ground through 600-grit paper and, finally, attack-polished on silk with 0.3-micron gamma alumina in a chromic-phosphoric-sulfuric acid solution.* Hardnesses were measured at room temperature on a standard Vickers hardness unit with a 2.5-kg load. Five impressions were made on each specimen and the results averaged. For both the sintered sleeves and the tubing, the measured surfaces were transverse to the long axis.

For microhardness measurements on re-extruded tubing, the specimens were first mounted in Bakelite. One of the surfaces transverse to the long axis was then metallographically polished and etched (see Section 5 p. 47). Finally, hardnesses were measured on a Tukon Hardness Tester with a 500-gram load.

4. Annealing

Annealing treatments on ring specimens from extruded or drawn tubing were carried out in a tantalum-element vacuum furnace. Pressures during heating and holding at temperature were always below 10^{-4} torr. Temperatures up to 1400°C (2550°F) were measured by means of a Pt/Pt-10% Rh thermocouple attached to the specimen support; temperatures above 1400°C were measured by means of a calibrated optical pyrometer.

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*The following stock solution diluted 10:1: 500 ml H_2O, 100 gms CrO_3, 70 ml conc. H_3PO_4, and 5 ml conc. H_2SO_4.

5. Metallographic Examination

Specimens were prepared for metallographic examination as follows: The specimens were mounted in Bakelite or stainless steel clamps. The mounted specimens were then wet ground through 600-grit paper and attack-polished on silk with 0.3-micron gamma alumina in a chromic-phosphoric-sulfuric acid solution. At this stage, the worked surface layer was removed by a two-second etch in Murakami's reagent. Final polishing was done on silk and nap wheels with 0.1-micron gamma alumina in the chromic-phosphoric-sulfuric acid solution.*

6. Density

Densities were measured by Archimedes (displacement) methods. The reported values have an uncertainty of ± 0.01 gm/cc.

C. Results and Discussion

1. Extrusion Sleeves

Fifty sintered tungsten-rhenium extrusion sleeves were evaluated during this year's program. Thirty-three sleeves were produced by the Bureau of Mines (Albany, Oregon) and seventeen were obtained from a commercial source. Fabrication of the Bureau of Mines sleeves is described in their Quarterly Metallurgical Progress Reports.** Hardness and density data for these sleeves are presented in Table 12; rhenium contents reported by the source, nominal sleeve dimensions and extrusion billet numbers are included in the table for reference.

Examination of Table 12 reveals that the Bureau of Mines sleeves have densities in the range 96 to 99 percent of the theoretical density and the Vendor A sleeves have densities in the range 95 to 98. Thus, sleeves from both sources are well above the 92.3 percent of theoretical density specified for W - 25 Re extrusion sleeves (NMI-1264, Appendix I). Hardness of the sleeves follows the densities, the higher hardnesses being associated with the higher densities.

Analyses supplied by the source show that the rhenium contents of one Vendor A sleeve (No. 200) and one Bureau of Mines sleeve (No. 212) were slightly below the allowed minimum, 24.5 w/o. The lower rhenium content of these sleeves was not regarded as sufficient cause for rejection. Both were successfully extruded to sound tubing (Tubes 200 and 217 respectively) which exhibited normal behavior during subsequent drawing

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*See footnote, p. 46.

**USBM-RC-1122, 1135, 1150 and 1158.

operations. Hence, there was no evidence that the slight rhenium deficiency of these sleeves had any effect on the properties.

Metallographic examination of sleeves failed to reveal significant differences in microstructures, either among sleeves from the same source or among sleeves from the two sources. Typical microstructures are illustrated in Figure 5. All of the sleeves had the characteristic equiaxed structures of material sintered at high temperatures. Grain sizes were relatively uniform, between ASTM Grain Size Nos. 6 and 7. Minor variations noted in pore structure were not considered significant. Past experience has shown that neither extrusion behavior nor quality of extruded tubing can be related to such differences. There was no evidence of second-phase particles in any of the sleeves.

Evaluations on the current sleeves show that they are uniform and satisfactory in quality. The quality of these sleeves is the same as the quality of earlier sleeves reported in NMI-1264.

2. <u>Extruded Tubing</u>

a. <u>Primary Extrusions</u>

The most extensive evaluations were performed on three tubes (Nos. 188, 207 and 211) produced from sintered sleeves. Evaluations on the remaining primaries were confined to routine metallographic examinations of their as-extruded structures. In addition to these evaluations, series of tests were carried out on primary extruded tubing (178-2 and 179-2; 198 and 199) to establish the effect of annealing on ring compression behavior. The purpose of these tests was to find a heat treatment that would produce softer and more ductile structures for drawability tests.

Results of ring compression tests on primary extruded tubing are given in Tables 13 through 15. Table 16 summarizes RCTT values derived from these results. RCTT (ring compression transition temperature) is defined as the lowest temperature for 50 percent of maximum deflection before failure. This parameter has been adopted in this program to provide a method for comparing the behavior of W-Re tubing. Microstructures of primary extruded tubing may be found in Figures 6 through 8.

In the following discussion, results for the more thoroughly evaluated tubes and for the annealing tests are treated individually. Results for the remaining tubes, because their evaluations were restricted to metallographic examinations, are discussed in a single section.

(1) <u>Tube 188</u>. Tube 188 was the first of its size, nominal 1/4-inch OD by 20-mil wall, extruded at 1600°C with a graphite core. Up to that time, only reference-size tubing had been extruded at 1600°C with a graphite core. It was of interest therefore to determine whether the graphite core had any effect on properties of the smaller tubing.

Results of the ring compression test on Tube 188, Table 13, show that this tube has low compression ductility at temperatures up to 600°C; the RCTT was 660°C. The ductility of this tube is markedly improved by a one-hour vacuum anneal at 1800°C. Apparently the RCTT for the annealed tubing was below 300°C, but its value could not be established definitely. Annealed specimens tested at 300 and 400°C failed at greater than 50 percent of maximum deflection. This apparent decrease in ductility at the higher temperatures was due to the fact that the 500 and 600°C test specimens fractured without the usual sharp drop in load; therefore the point of fracture could not be determined accurately on the load-deflection curves.

The effect of the graphite core on ring compression ductility may be assessed from a comparison of RCTT values in Table 16 for Tube 188 and Tube 177, an earlier tube of the same size that was extruded with an all-molybdenum core. (Ring compression results for Tube 177 may be found in NMI-1264, Table 41). Such a comparison shows the absence of any effect due to the graphite core. Tubes 188 and 177 have comparable RCTT values in both the as-extruded and 1800°C annealed conditions.

The microstructures of Tube 188 (not shown) were also unaffected by the graphite core. In both the as-extruded and 1800°C annealed condition, the structures were almost identical to those of Tube 177 and of reference-size tubing (Figure 7). As-extruded Tube 188 had a wrought banded structure with a few narrow bands of fine-grained recrystallized material. The 1800°C structure was fully recrystallized and equiaxed. Spherical second-phase particles, forming stringers in the extrusion direction, were observed in the as-extruded and recrystallized specimens. The amount of second phase in both cases was small, certainly no greater than observed in other primary extruded tubes.

The evaluations on Tube 188 demonstrated that the graphite core did not influence properties of 1/4-inch OD by 20-mil tubing. Tube 188 was comparable in all respects with tubing of the same size extruded with an all-molybdenum core and with reference-size tubing extruded with a graphite core.

(2) Tube 207. Tube 207 was an oversize 1/4-inch OD by 20-mil wall tube. The extruded dimensions of this tube were selected to permit about a 30 percent reduction during subsequent drawing to the final 1/4 x 20 size. The evaluations on Tube 207 were performed to confirm the extrusion step in the fabrication process for 1/4 x 20 tubing.

The ring compression results for Tube 207, Table 13, showed that the behavior of this tube was typical of extruded W-Re tubing. Its ductility was relatively low in the as-extruded condition; the RCTT values for both front and rear were above 700°C, the highest test temperature used for the ring-compression tests. The one-hour anneal at

$1800^{\circ}C$ brought about a marked improvement in ductility. The RCTT in this condition, as shown in Table 16, was about $300^{\circ}C$.

The microstructures of Tube 207 before and after annealing are illustrated in Figure 6. These structures are characteristic of $1600^{\circ}C$ extruded W-Re tubing. The as-extruded structure is heavily wrought and contains a relatively small amount of recrystallized material; the $1800^{\circ}C$ annealed structure is fully recrystallized. Second-phase particles can be seen in both structures. The particles in the $1800^{\circ}C$ annealed specimen appear to be larger and more spherical indicating that the particles agglomerated during the annealing treatment.

(3) Tube 211. Tube 211 was the first produced from a Bureau of Mines powder metallurgy sleeve. Because of its long length (the sleeve was 8 inches long) this tube was sampled in the center as well as in the front and rear.

As shown by the ring compression results in Table 13, as-extruded Tube 211 exhibited a large front-to-rear difference in behavior. The fractions of maximum deflection at failure of specimens from the center and rear were almost twice those of specimens from the front. The one-hour anneal at $1800^{\circ}C$ improved ductility and eliminated the front-to-rear difference in behavior. The test results indicated that the RCTT for the annealed tubing was 360 to $390^{\circ}C$. However, these values could not be established because specimens tested at $600^{\circ}C$ failed at less than 50 percent of maximum deflection. The apparent decrease in ductility of the $600^{\circ}C$ specimens was caused by the development of sharp bend radii on the inside of the specimens in the area in contact with the platens.

A comparison of the microstructures of Tube 211, Figure 7, with those of Tube 207, Figure 6, shows that structures of the two tubes are almost identical. The only significant difference is in the amount of second phase. There are only a few widely scattered particles in the structure of Tube 211 made from the Bureau of Mines sleeve.

(4) Stock Tubing. Tubes 226 through 230 provided stock for drawing to 1/4 x 20; Tubes 236 to 241 were extruded from reference-size sleeves, primarily for re-extrusion stock.

Evaluations on Tubes 226 through 230 were limited to metallographic examination in order to conserve material for drawing. Tubes 226 and 227 were sampled in four locations: front, rear and one-third from each end. Tubes 228, 229 and 230 were sampled at the front and rear.

Photomicrographs of Tube 226, Figure 8, illustrate the microstructures of this batch of tubes. All five tubes exhibited somewhat greater amounts of recrystallization than usually observed in $1600^{\circ}C$ primary extruded W-Re tubing. (See Figures 6 and 7 for typical as-extruded

structures). These tubes also had larger front-to-rear differences than usual, except for Tube 227, which had a uniform structure over its entire length. The difference in this set of five tubes may be a result of its being extruded at higher speeds than usual (see Table 1).

Tubes 236 and 237 were examined for microstructure. These tubes were nominal 0.3-inch diameter by 0.030-inch wall tubing produced in 25:1 reduction extrusions at 1600°C. The metallographic examinations revealed that both tubes have wrought fibrous structures interspersed with a few narrow bands of fine-grained recrystallized material, and are quite free of second phase. These structures are characteristic of 1600°C primary extrusion tubing.

The remaining tubes (Tubes 238, 239, 240 and 241) were extruded at a 16:1 reduction and were not evaluated.

b. Annealed Tubing

Two series of tests on the effect of annealing on ring compression behavior were intended to guide the selection of a heat treatment to improve drawability.

(1) Tubes 178-2 and 179-2. The purpose of the ring compression tests on Tubes 178-2 and 179-2, 3/8-inch diameter reference process tubing, was to examine the effects of annealing on ductility in greater detail than had been done previously. Earlier work (NMI-1264, Table 42) indicated that ductility of extruded tubing was improved by annealing at 1400 and 1800°C and that the degree of improvement increased with increasing temperature of anneal.

Results are given in Table 14 for specimens of Tubes 178-2 and 179-2 tested in the as-extruded condition and after annealing one hour at 200°C intervals between 1000 and 1800°C. These results indicate that the apparent effect of annealing on the ductility depends on the test temperature and differs somewhat for the two tubes.

The ring compression behavior of both tubes at the two test temperatures does not respond as favorably to annealing at 1800°C as tubing examined earlier. No reason can be given for this difference in behavior. Possibly it is related to the fact that Tubes 178-2 and 179-2 were produced by tandem extrusion. However, metallographic examination failed to reveal significant differences between their microstructures and those of other 1600°C extruded tubing, either in the as-extruded or annealed conditions. The RCTT values of the various tubes in the as-extruded condition are quite similar.

(2) Tube 199. The purpose of the ring compression tests on Tube 199 was to guide the selection of a predraw heat treatment that would soften extruded W-Re tubing. Draw test results which were

available at that time indicated lower draw forces would reduce problems of point breakage and mandrel necking. Accordingly, specimens from the center of Tube 199 were annealed 15, 30 and 60 minutes at 1700 and 1800°C. The annealed specimens were then ring compression tested at 400 and 500°C (750 and 930°F) near the drawing temperatures. As-extruded specimens were also tested for comparison.

Results of the ring compression tests on Tube 199 are given in Table 15. As may be seen in the table, both as-extruded specimens failed at less than 0.025-inch deflection; therefore, no direct comparison could be made between strengths of as-extruded and annealed specimens. However, the load at fracture of the as-extruded specimens indicated that they were substantially stronger than the annealed specimens. Of the two temperatures investigated, the higher temperature, 1800°C, reduced the load more. The normalized loads at 0.025-inch deflection for the 1800°C annealed specimens are about 100 lbs. lower than the corresponding loads for the 1700°C annealed specimens.

Results in Table 15 also show that all of the annealed specimens had greater compression ductility than the as-extruded specimens. Annealed specimens failed at 20 to 50 percent of maximum deflection whereas as-extruded specimens failed at 6 to 8 percent of maximum deflection. In contrast to their strength properties, ductility of the annealed specimens was independent of annealing conditions (for the times and temperatures investigated) and the test temperature.

Metallographic examinations of companion specimens annealed with the ring compression specimens revealed significant differences in microstructure. Specimens annealed 15 and 30 minutes at 1700°C were partially recrystallized, the 15-minute specimen about 75 percent and the 30-minute specimen about 90 percent. The specimen annealed 60 minutes at 1700°C was fully recrystallized, but had a duplex grain size and a tendency for grains to be aligned in the extrusion direction. All specimens annealed at 1800°C were fully recrystallized, the grain size increasing with increasing time of anneal. These differences in microstructure explain the strength differences of the 1700 and 1800°C annealed ring compression specimens.

In light of these results, an anneal of 15 minutes at 1800°C was recommended prior to drawing of as-extruded tubing.

(3) Tube 198. Based on the results for Tube 199 described above, 27 inches of Tube 198 was annealed 15 minutes at 1800°C in an A-15H_2 atmosphere (Table 15, page 26) by self-resistance heating. Table 15 presents results of ring compression tests on specimens from the center section of the annealed tube.

Tube 198 was not ring compression tested in the as-extruded condition and, hence, the effect of the 15-minute 1800°C anneal cannot be

evaluated quantitatively. The similarity of the results for Tube 198 and 199 in the annealed condition indicates that the anneal accomplished significant softening of Tube 198, as was found for Tube 199. Normalized loads at 0.25-inch deflection for Tube 198 are 26 and 75 lb./in. greater at 400 and 500°C, respectively, than those for 15-minute 1800°C annealed Tube 199. However, the anneal failed to produce the expected improvement in ductility of Tube 198. The fractions of maximum deflections at failure of the Tube 198 specimens are about half those for Tube 199 specimens.

Metallographic examination of Tube 198 showed that the 15-minute anneal at 1800°C produced a fully recrystallized, equiaxed structure. However, as shown by the photomicrographs in Figure 9 , the grain size of Tube 198 was significantly larger than that of Tube 199 annealed one hour at 1800°C. In addition, Tube 198 contained more second phase than was observed in any other annealed W-Re tubing, except possibly Tube 199. Most of the second phase was in the form of spherical particles which formed "stringers" aligned in the extrusion direction.

c. Secondary Extrusions

In the evaluations on secondary extrusions, five tubes were examined extensively. Three of these tubes (Nos. 171, 182 and 186) were extruded with all-molybdenum cores and two (Nos. 223 and 224) were extruded with $MoCaC_2$ cores. Tubes 232 through 235 from Series Q were examined metallographically. In addition to these evaluations, hardness measurements and microprobe analyses were performed on re-extruded tubing and the stock tubing used for the re-extrusions. Results of these evaluations are described below.

(1) Tube 171. Ring compression tests on Tube 171, 0.13-inch OD by 0.010-inch wall tubing produced by secondary extrusion at 1200°C, were the first such tests carried out on small-diameter, thin-wall tubing. Techniques developed for the reference 3/8-inch OD tubing were used for specimen preparation and testing.

Results of the ring compression tests on Tube 171 are presented in Table 17. In the tests, the ring specimens behaved in the same manner as larger diameter specimens. Fracture was accompanied by a sharp drop in load and, hence, the ductility at fracture could be readily determined.

The results in Table 17 show that Tube 171 does not have high ductility in the as-extruded condition. This lack of ductility is not surprising because the stock for this tube, a section of as-extruded Tube 164 (a 1600°C primary extrusion), had low ring compression ductility. Furthermore, tests on tubing from the earlier extrusion temperature series, (see Table 42, NMI-1264), indicated that the as-extruded ductility of W-Re tubing decreased with decreasing extrusion temperature. A 1200°C (2200°F) primary extruded tubing (Tube 118D) had an RCTT value above 700°C (1290°F).

Metallographic examinations on Tube 171 revealed the following: In the extruded condition, the structure is heavily fibered with the fibers aligned in the extrusion direction. Annealing one hour at 1400 or 1500°C causes a general coarsening of the fibrous structure and a small amount of recrystallization. The structure is fully recrystallized following a 1600°C anneal but the grains have very irregular shapes and tend to be aligned in the extrusion direction. Both the 1700 and 1800°C anneals produce equiaxed structures. The microstructures of Tube 171 before and after the various anneals are almost identical with those of Tube 182 in Figure 10.

(2) Tube 182. Table 17 includes the results of ring compression tests on Tube 182. This tube was produced by secondary extrusion at 1200°C (2200°F); the nominal extrusion reduction was 25:1. The stock material was a section of Tube 178-2, a 1600°C (2910°F) primary extrusion.

One of the aims of the ring compression tests on Tube 182 was to determined whether a heat treatment would facilitate drawing of small-diameter tubing. Accordingly, specimens were tested in the as-extruded condition and four heat-treated conditions: (1) stress-relieved one hour at 1000°C (low-temperature stress relief); (2) stress-relieved one hour at 1400°C (high-temperature stress relief); (3) recrystallized one hour at 1600°C, and (4) recrystallized one hour at 1800°C. Examination of the results in Table 17B shows that the 1400°C stress relief and the 1600°C and 1800°C recrystallization anneals produced large improvements in the ductility of Tube 182. Each of these heat treatments lowered the RCTT from above 700°C to about 200°C. The 1000°C stress relief failed to improve the ductility of Tube 182. Based on these results, it appears that a high-temperature stress relief or a recrystallization anneal could be used if necessary to improve the drawability of re-extruded small-diameter W-Re tubing.

Photomicrographs of as-extruded and annealed specimens of Tube 182 are reproduced in Figure 10. These photomicrographs show that the as-extruded specimen has a fibrous highly wrought structure with the fibers aligned in the extrusion direction. The structure is unchanged by the 1000°C anneal. Both the 1400°C and the 1500°C annealed specimens show general coarsening of the structure and a few small recrystallized grains at the fiber boundaries. The 1600°C annealed specimen is fully recrystallized but the grains have irregular shapes and are elongated in the extrusion direction. The 1700 and 1800°C annealed specimens have coarser and more equiaxed grain structures.

As stated earlier, the microstructures of Tubes 171 and 182 are almost identical in both the as-extruded and annealed conditions. The annealed structures are also very similar to those of Tube 118D, a 1200°C primary extrusion (see NMI-1264, Figure 37; also NMI-2125, Figure A-2, p. 33-34.) Thus it appears that annealing behavior of 1200°C re-extruded tubing is insensitive to extrusion reduction ratio and is not affected by prior extrusion at 1600°C.

However, there are differences in the as-extruded structures of Tubes 171, 182 and 118D. Tubes 171 and 182 show evidence of greater amounts of cold work than Tube 118D. This higher degree of cold work undoubtedly is due to the fact that Tubes 171 and 182 received a prior reduction of 16:1 at 1600°C before the 9:1 and 25:1 reduction, respectively, at 1200°C.

The one-hour recrystallization temperature of Tubes 171 and 182 is about 100°C below that of tubing produced in primary extrusions at 1600°C. Tubes 171 and 182 are fully recrystallized after a one-hour anneal at 1600°C whereas a one-hour anneal at 1700°C is required to recrystallize the 1600°C primary extrusions. The primary extrusions exhibit appreciable amounts of cold work after a one-hour anneal at 1600°C. Thus it appears that the additional cold work introduced by a 9:1 or 25:1 reduction at 1200°C has an appreciable effect on recrystallization behavior.

(3) Tube 186. Evaluations on Tube 186 paralleled those on Tube 182 since both tubes were re-extruded with a 25:1 reduction at 1200°C. As in the case of Tube 182, the stock material for Tube 186 was a section of as-extruded Tube 178-2.

Results of ring compression tests on Tube 186, Table 17C, show that this tube has the same response as Tube 182 to the 1400°C stress relief and 1800°C recrystallization anneals. Both heat treatments lowered the RCTT by about 500°C. Like Tube 182, Tube 186 had low ring compression ductility in the as-extruded condition. Tube 186 was not tested in either the 1000°C or 1600°C heat-treated conditions. However, it is reasonable to assume that these heat treatments would have produced the same effects as they did in Tube 182.

Metallographic examinations on Tube 186 revealed that its as-extruded and 1800°C annealed structures differed from those of Tube 182 (Figure 10). Although Tube 186 had a wrought structure in the as-extruded condition, the degree of cold work appeared to be far less than that in Tube 182. The fibrous bands in Tube 186 were much wider and there were a few widely scattered areas of recrystallized material. After a one-hour anneal at 1800°C, Tube 186 had a significantly larger grain size than Tube 182. No reasons can be given for the microstructural variations in these tubes, which were re-extruded under the same conditions and from the same stock material.

(4) Tube 223. Tube 223 was re-extruded at 1200°C with a nominal reduction of 16:1. The stock tubing was a recrystallized piece of Tube 199 which had received a 2 percent drawing reduction to improve dimensions (Draw Test 137A). Tube 223 also differed from the tubes discussed above in that it was re-extruded with a Mo-CaC_2 core rather than an all-molybdenum core.

As may be seen in Table 18, Tube 223 had low ring compression ductility in the as-extruded condition, fracturing at less than 15 percent of maximum deflection at temperatures up to 700°C. This behavior is very much like that of Tubes 182 and 186 which were produced in 1200°C re-extrusions of as-extruded Tube 178-2. Hence, no special significance may be given to the low ring compression ductility of as-extruded Tube 223.

The as-extruded and 1800°C-annealed microstructures of Tube 223 are shown in Figure 11. The as-extruded structure is highly worked without any evidence of recrystallization. The 1800°C-annealed structure is fully recrystallized. Several "stringers" and numerous minute particles may be seen in both structures. The stringers, which resemble elongated voids, are believed to indicate the prior presence of W-Re sigma phase particles that were removed during the metallographic preparation.

(5) Tube 224. Tube 224 was like Tube 223 in all respects except extrusion temperature; Tube 224 was re-extruded at 1500°C. In spite of this difference in extrusion temperature, the ring compression behavior and microstructures of Tube 224 are almost identical with those of Tube 223. As shown by the results in Table 18B, Tube 224 had low ring compression ductility in the as-extruded condtion. The specimens failed at less than 15 percent of maximum deflection at temperatures up to 700°C. The only difference in the microstructures of Tubes 223 and 224 was in the amount of second phase. Tube 224 appeared to have fewer "stringers" and minute particles.

Results for the five re-extruded tubes (Tubes 223 and 224 extruded with Mo-CaC_2 cores and Tubes 171, 182 and 186 extruded with all-molybdenum cores) indicate that as-extruded ring compression behavior is not influenced by extrusion temperature or by a prior recrystallization anneal of the re-extrusion stock. These parameters also appear to have little effect on as-extruded structure and annealing behavior. Only Tube 186 had microstructures that differed from those of the other tubes.

(6) Series Q Tubing. Specimens for metallographic examinations on Tubes 232 through 235 (Series Q) were taken from sound portions of the tubes as far removed as possible from any visible defects (p. 14). These examinations revealed no differences in structure of the tubes although Tubes 232 and 233 were extruded at 1200°C and Tubes 235 and 235 were extruded at 1500°C. In each case, the structure was fully wrought without any evidence of recrystallization. Microstructures of these tubes were almost identical to that of Tube 223 illustrated in Figure 11 with respect to the matrix structure and the number of stringers and fine particles.

The metallographic examinations failed to reveal any structural features that could account for the unsatisfactory extrusion results.

(7) Hardness Measurements. Hardness measurents were carried out on re-extruded tubing and on stock tubing used for the re-extrusion. Purpose of these measurements was to determine whether a relationship existed between hardness and re-extrusion results. All hardness specimens were mounted in Bakelite, metallographically polished and etched with Murakami's reagent. Hardnesses of re-extruded tubes were measured on a Tukon Hardness Tester with a 500-gram load; those on stock tubing were measured on a standard Vickers hardness unit with a 2.5-kg load. In all cases, the measured surfaces were transverse to the long axis of the tubes.

Results of the hardness measurements are summarized in Table 19. It is apparent from this table that the relation between hardness of the stock and the re-extruded tubing is different for the two re-extrusion temperatures. In the case of the 1200°C re-extrusions, annealing of the stock material has only a small softening effect on the re-extruded tube (Tubes 213 and 214 vs. 232). In the case of the 1500°C re-extrusions, however, tubing produced from recrystallized stock is far softer than that produced from as-drawn stock (Tubes 224 and 235 vs. 215B).

The hardness measurements failed to demonstrate a relationship between hardness and tube quality. Both sound and unsound tubes show about the same variation in hardness before or after extrusion.

(8) Microprobe Analyses. As a further check on the quality of stock used for re-extrusion, electron microprobe analyses were run to determine whether rhenium segregation contributed to re-extrusion results. Tubes 178-2 and 217, and the sintered sleeves from which they were produced, were selected for these analyses because re-extrusions of Tube 178-2 produced sound tubing (Tubes 182, 186 and 190) whereas those of Tube 217 did not, the tubes containing chevron tears and other surface defects (Tubes 232-235, from Draw 157A).

The microprobe analyses indicated that the rhenium content was uniform throughout the samples of extruded tubes and sintered sleeves. (The uncertainty of the probe analysis is estimated to be $\pm$ 2 w/o Re). There was no evidence of microsegregation or sigma phase particles. Furthermore, the rhenium contents were at the expected level, about 25 w/o. These results showed that the re-extrusion results could not be attributed to segregation or differences in rhenium contents of the stock materials.

3. Drawn Tubing

Photomicrographs in Figure 12 show the microstructural changes that occurred during drawing of Tube 224, a 1500°C re-extrusion with a Mo-CaC_2 core. This tube was successfully drawn (Draw Test 166) to 30.8 percent cumulative reduction in four passes at 510°C. The work piece was annealed 1/2 hour at 1400°C after each pass. The photomicrographs show that the extent of recrystallization increased progressively

after each drawing and annealing step. The structures exhibit varying numbers of dark spots which are believed to be etch pits. Examinations at magnifications up to 2000 diameters showed most of these spots had triangular shapes typical of etch pits rather than the rounded shapes that would be expected for second-phase particles.

Tube 223, a 1200°C re-extrusion with a Mo-CaC_2 core, showed the same sequence of structural changes as Tube 224 illustrated in Figure 12. Tube 223 was drawn (Draw Test 153) to 28.8 percent cumulative reduction in five passes at 510°C. Intermediate anneals of 1/2-hour at 1400°C were used after each pass. Photomicrographs of Tube 223 were shown in NMI-2136, Figure A-3, p. 33. The only difference in structures of the two drawn tubes was in the number of dark spots. These spots were more numerous in Tube 223 and they appeared to increase with increasing reduction. Apparently, Tube 223 had a greater tendency for etch-pitting than did Tube 224.

TABLE 1

CONDITIONS FOR TUNGSTEN - 25 $^{w}/o$ RHENIUM PRIMARY EXTRUSIONS

Billet No.	Sleeve No.	Reduction (%) From Tool Sizes	Reduction (%) From Tubing Diameter	Extrusion Speed (ipm)	Extrusion Force (tons)	Extrusion Pressure (tsi)	Extrusion K (tsi)	Intended Use
188	S192	16	15.4	300	400	55	20	Test use of graphite for 1/4-20 tubing.
195	S208	15.5	15.3	90	425	58.5	16	For drawing to 1/4-20.
196	S209	16	15.9	100	450	61.9	22.3	
207	S210	16	15.5	100	510	70.3	25.4	
218	S213	14.4	14.4	300	395	54.4	19.7	
226	S214	16	15.8	420	350	48.1	17.4	
227	S215	16	15.1	390	385	53.0	19.2	
228	S216	16	14.4	420	380	52.4	18.9	
229	S217	16	14.7	450	340	46.8	16.5	
230	S218	16	14.5	420	365	50.3	18.2	
242	S219	16	15.7	240	420	57.9	20.9	
243	S220	16	15.6	240	410	56.5	20.4	
244	S221	16	16.0	300	395	54.4	19.6	
245	S222	16	15.8	240	415	57.1	20.6	
193	S196	16	--	90	480	66.0	18.0	Mainly re-extrusion stock; some used for drawing.
194	S197	16	--	90	440	60.5	17.0	
197	S182	16	15.5	100	480	66.0	23.8	
198	S198	16	15.7	100	455	62.6	22.6	
199	S199	16	16.0	100	510	70.4	25.4	
200	S200	25.4	21.7	100	600	82.5	25.6	
201	S193	24.8	23.0	100	Stalled			
202	S194	25	23.1	250	565	77.9	24.2	
211	S211	16	15.5	250	475	65.4	23.6	
217	S212	16	15.4	350	465	62.6	22.6	

(Table continued on next page)

TABLE 1 (CONTINUED)

Billet No.	Sleeve No.	Reduction (%) From Tool Sizes	Reduction (%) From Tubing Diameter	Extrusion Speed (ipm)	Extrusion Force (tons)	Extrusion Pressure (tsi)	Extrusion K (tsi)	Intended Use
238	S225	16	--	240	488	67.1	24.2	Mainly re-extrusion stock; some used for drawing.
239	S226	16	14.9	270	475	65.5	23.6	
240	S227	16	15.2	270	475	65.5	23.6	
241	S228	16	14.9	240	490	67.5	24.4	
189	S203	25	23.4	100	830	84.0	26.0	Odd size (BM arc-cast sleeve) in 3.550 liner.
236	S223	25	23.5	No Record				For delivery to ANL.
237	S224	25	23.5	180	545	75.0	23.3	

NOTES

A. Tooling

1. Liner

Size: 3.050-inch, in 1400-ton press

Temp: 900°F

2. Die

Type: Internal, integral, ZrO_2 coated

Temp: Ambient

Lubricant: Glass

B. Billet heating time: 10 min. + 5 min. soak

C. All billets extruded at 1600°C with glass lubrication.

TABLE 2

DIMENSIONS (INCHES) OF TUNGSTEN - 25 $^{w}/_{o}$ RHENIUM PRIMARY EXTRUDED TUBING

Tube No.	Sleeve[1]						Tube					
	No.	OD		Wall		Nominal Length	Length Evaluated	Posi-tion	OD		Wall	
		Max.	Min.	Avg.	Range				Max.	Min.	Max.	Min.
T188	S192	1.000	0.999	0.795	0.001	4	36	-	0.259	0.253	0.024	0.022
T195	S208	1.090	1.089	0.077	0.002	8	88	-	0.288	0.270	0.024	0.021
T196	S209	1.117	1.116	0.085	0.001	8	58	F	0.284	0.275	0.024	0.023
								R	0.286	0.275	0.024	0.023
T207	S210	1.137	1.136	0.097	0.002	4	42	F	0.292	0.282	0.028	0.026
								R	0.300	0.283	0.028	0.026
T218	S213	1.081	1.079	0.075	0.001	8	80	F	0.286	0.284	0.024	0.023
								M	0.285	0.283	---	---
								R	0.285	0.283	---	---
T226	S214	1.133	1.131	0.100	0.002	8	88	F	0.292	0.286	0.029	0.028
								M	0.294	0.286	0.030	0.029
								R	0.299	0.285	0.031	0.029
T227	S215	1.139	1.134	0.101	0.003	8	84	F	0.302	0.296	0.029	0.028
								M	0.298	0.293	0.029	0.028
								R	0.298	0.290	0.031	0.030
T228	S216	1.137	1.135	0.102	0.001	8	90	F	0.298	0.296	0.031	0.028
								M	0.304	0.297	0.007	---
								R	0.301	0.297	0.033	0.032

(Table continued on next page)

TABLE 2 (CONTINUED)

Tube No.	Sleeve[1]						Tube					
	No.	OD		Wall		Nominal Length	Length Evaluated	Position	OD		Wall	
		Max.	Min.	Avg.	Range				Max.	Min.	Max.	Min.
T229	S217	1.114	1.112	0.089	0.002	8	92	F	0.295	0.289	0.026	0.024
								M	0.298	0.287	---	---
								R	0.295	0.285	0.030	0.028
T230	S218	1.136	1.132	0.099	0.002	8	90	F	0.298	0.296	0.031	0.029
								M	0.295	0.290	---	---
								R	0.301	0.293	0.032	0.031
T242	S219	1.136	1.135	0.094	0.000	8	92	F	0.289	0.286	---	---
								M	0.287	0.284	---	---
								R	0.290	0.287	---	---
T243	S220	1.136	1.135	0.095	0.000	8	100	F	0.294	0.288	---	---
								M	0.290	0.285	---	---
								R	0.294	0.288	---	---
T244	S221	1.116	1.115	0.090	0.000	8	96	F	0.283	0.281	---	---
								M	0.278	0.275	---	---
								R	0.281	0.277	---	---
T245	S222	1.137	1.136	0.1015	0.001	8	102	F	0.288	0.283	---	---
								M	0.287	0.283	---	---
								R	0.288	0.286	---	---
T193	S196	1.500	1.500	0.160	0.001	4	30	-	0.500[2]	0.409	---	---
T194	S197	1.500	1.500	0.160	0.001	4	42	-	0.391	0.364	0.044	0.042
T197	S182	1.500	1.500	0.1595	0.001	4	32	-	0.389	0.373	---	---

(Table continued on next page)

TABLE 2 (CONTINUED)

Tube No.	Sleeve[1]						Tube					
	No.	OD		Wall		Nominal Length	Length Evaluated	Position	OD		Wall	
		Max.	Min.	Avg.	Range				Max.	Min.	Max.	Min.
T198	S198	1.500	1.500	0.1605	0.001	4	38	-	0.388	0.369	0.045	0.043
T199	S199	1.501	1.500	0.161	0.001	4	38	-	0.387	0.368	0.044	0.043
T200	S200	1.500	1.500	0.160	0.001	4	58	F	0.342	0.311	0.042	0.039
								R	0.332	0.310	0.040	0.038
T201	S193	1.800	1.799	0.149	0.0005	4	28	F	0.379	0.373	0.043	0.039
								R	0.379	0.370	0.035	0.034
T202	S194	1.800	1.799	0.147	0.003	4	62	F	0.377	0.369	0.034	0.033
								R	0.382	0.373	0.039	0.037
T211	S211	1.423	1.420	0.113	0.003	8	102	F	0.363	0.358	0.034	0.032
								M	0.365	0.358	0.033	0.032
								R	0.366	0.358	0.032	0.030
T217	S212	1.507	1.506	0.171	0.002	8	92	F	0.385	0.382	---	---
								M	0.384	0.380	0.046	0.045
								R	0.385	0.383	0.002	---
T238	S225	1.501	1.500	0.159	0.002	4	knobs and depressions - see text					
T239	S226	1.500	1.500	0.158	0.004	4	38	F	0.384	0.382	0.042	0.041
								M	0.391	0.388	---	---
								R	0.390	0.386	---	---
T240	S227	1.499	1.499	0.160	0.001	4	38	F	0.385	0.382	---	---
								M	0.384	0.381	---	---
								R	0.388	0.385	---	---
T241	S228	1.501	1.500	0.159	0.005	4	36	F	0.383	0.380	---	---
								M	0.385	0.378	---	---
								R	0.390	0.382	---	---

(Table continued on next page)

TABLE 2 (CONTINUED)

Tube No.	Sleeve[1]						Tube						
	No.	OD		Wall		Nominal Length	Length Evaluated	Position	OD		Wall	
		Max.	Min.	Avg.	Range				Max.	Min.	Max.	Min.
T189	S203	1.858	1.861	0.260	0.004	2	27	F M R	0.445 0.374 0.428	0.436 0.371 0.421	--- --- ---	--- --- ---
T236	S223	1.501	1.500	0.159	0.005	4	60	F M R	0.309 0.308 0.313	0.306 0.307 0.309	0.037 --- 0.036	0.036 --- 0.035
T237	S224	1.500	1.500	0.159	0.005	4	60	F M R	0.310 0.311 0.312	0.307 0.308 0.310	0.036 --- 0.036	0.035 --- 0.035

(1) Sleeve 182 from Vendor C.
Sleeves 192, 208-210, 223-228 from Vendor A.
Remainder from Bureau of Mines.

(2) Die washed.

TABLE 3

CONDITIONS FOR TUNGSTEN - 25 $^{W}/_{o}$ RHENIUM RE-EXTRUSIONS

Tube No.	Stock Tube No.	Heat Treat-ment	CaC_2 Core: Pre-Extruded or Tamped	CaC_2 Core: Dia. (in.)	Temp (°C)	Reduction (%): From Tool Sizes	Reduction (%): From Tube Diameter	Extrusion: Speed (fpm)	Extrusion: Force (tons)	Extrusion: Pressure (tsi)	Extrusion: K* (tsi)	Factor Investigated	Results
T180	174	None	Solid Mo	.290	1200	24.5	20	65	250	76.5	24	W tubes, Mo core. Test feasibility with metal core and higher reductions.	Good tubes
T181						36.0			Stall				
T184						24.4	24.4	90	405	55.7	17		
T185						36.0	38.6	90	485	66.7	19		
T182	178	None	Solid Mo	.290	1200	24.5	20.5	65	310	92	29	Reduction	Good tube
T186						25	22.7	90	420	57.8	18		Good tube
T187						36	45	90	450	62.2	17		Chevron tears
T190	178	None	T	.201	1200	20	23.5	100	385	53	18	Core diameter	Good tube
T191				.221		20	23.0	90	370	51	17		10 pieces
T192				.250		20	--	90	400	55	19		30 pieces
T205	194	None	P	.20	1200	19.4	--	100	360	49.5	17.9	Reduction	6 pieces
T206						16.0	15.2	100	385	53.0	19.2	Confirm T190	3 pieces
T209	194	1450°C	T	.201	1200	16.0	--	100	320	44	15.9	Tamped vs. pre-extruded CaC_2; S-R anneal	Many pieces
T210			P	.17		16.0	15.1	100	312	43	15.5		3 tears
T212	199	None	P	.17	1200	16.0	14.9	90	340	46.8	16.9	Control	Tears
T213		1350°C			1200	16.0	14.7	90	330	45.5	16.4	S-R anneal	Tears
T214		1450°C			1200	16.0	15.0	90	335	46.1	16.7	S-R anneal	Tears
T215A		None			1300	16.0	--		Stall			T-(1300°C)	Good but undersize
T215B		None			1500	16.0	29	80	525	72.3	26.1	T-(1500°C)	Good
T216		None			1200	9.0	8.6		No record			R	Good
T220	199	None	P	.17	1200	16.0	--	65	225	68.9	24.8	Die diameter	Many pieces
T221	196	None		.15	1500	14.4	12.3	45	490	67.5	25.3	R, T, stock tube size	4 pieces
T222	196	None		.15	1200	14.4	13.6	90	305	42.0	15.7	R, stock tube size	2 pieces
T223	199	1800°C		.17	1200	16.0	14.8	90	320	44.0	15.9	Recrystallization anneal	Good
T224	199	1800°C		.17	1500	16.0	14.3	130	515	70.9	25.6	T, recrystallization anneal	Good
T225	217	None		.17	1500	16.0	13.6	60	510	70.1	25.3	T	Many tears

(Table continued on next page)

TABLE 3 (CONTINUED)

Tube No.	Stock Tube No.	Heat Treat-ment	CaC_2 Core: Pre-Extruded or Tamped	CaC_2 Core: Dia. (in.)	Temp (°C)	Reduction (%): From Tool Sizes	Reduction (%): From Tube Diameter	Extrusion: Speed (ipm)	Extrusion: Force (tons)	Extrusion: Pres-sure (tsi)	Extrusion: K* (tsi)	Factor Investigated	Results
T232 T233 T234 T235	217	1800°C	P	.17	1200 1200 1500 1500	16.0 20.0 16.0 20.0	15.2 18.1 14.9 17.6	90 90 150 No record	310 335 470	42.6 46.1 64.7	15.4 13.6 23.4	Control R T T, R	Tears Tears Tears Tears
T246	217 199 217	1800°C	P	.19	1200	16.0	15.0 -- --	90	350	48.2	17.4	Control	Good Many tears 2 tears
T247	217 239 217	1800°C	P	.19 .19 .15	1200	16.0	-- 15.8 15.6	90	365	50.2	18.1	Virgin Mo	1 tear 1 tear Good
T248	217 239 217	1800°C	P	.22 .19 .19	1200	9.0	8.6 -- 8.6	90	285	39.2	17.8	Virgin Mo, R	1 tear 1 tear Good
T249	236 237	1800°C	P	.15	1200	9.0	8.0 9.0	90	245	33.8	15.4	Virgin Mo, R, Stock tube size (.31")	Bumpy 1 tear
T250	217	1800°C	P	.19	1500	16.0	-- 14.2 15.0	180→ 15	500	68.9	24.8	Holding time in furnace	7 tears No defects Rough surface
T251	217 239 217	1800°C	P	.19 .15 .15	1200	16.0	-- -- 15.2	90	350	48.2	17.4	TZM inner filler	Bumpy 3 tears Good
T252 T253 T254 T255 T256	239	1800°C	T	.172	1200 1200 1525 1525 1200	16.0 16.0 16.0 16.0 16.0	-- 16.6 16.6 16.8 17.7	50 100 100 50 50	625 600 550 425 340	86.0 82.5 75.6 58.5 46.7	31.0 29.8 27.3 21.2 16.5	TZM, T, speed TZM TZM TZM, speed Speed	Broke up Broke up Bumpy Slight bumps Good

(Table continued on next page)

TABLE 3 (CONTINUED)

Tube No.	Stock Tube No.	Heat Treatment	CaC_2 Core: Pre-Extruded or Tamped	CaC_2 Core: Dia. (in.)	Temp (°C)	Reduction (%): From Tool Sizes	Reduction (%): From Tube Diameter	Extrusion: Speed (ipm)	Extrusion: Force (tons)	Extrusion: Pressure (tsi)	Extrusion: K* (tsi)	Factor Investigated	Results
T257		1800°C			1200	16.0	18.3	60	355	49.0	17.7	Dup. 256; larger core	Good
T258	239	1800°C	T	.201	1550	16.0	17.4	90→45	595	67.5	24.4	Dup. 255; larger core	Chevrons, incipient tears
T259		None			1235	16.7	17.9	50	220	67.5	24.0	Slow speed, 2-in. liner on 300-ton press	3 pieces
T260	239	1800°C	T	.201	1550	16.0	--	60	490	67.5	24.3	T, speed, Mo filler	Severe undulations; chevrons
T261					1235		15.6	50	265	81.1	29.2	Speed, press, with annealed tube	Good, 1 dimple
T262	240						--		310	42.6	15.4	None	4 chevron tears
T263	217						--		305	42.0	15.2	None	2 chevron tears
T264	240	1800°C	T	.201	1200	16.0	--	60	315	43.5	15.7	None	1 tear; 1 abrupt diameter change
T265	239						16.5		330	44.1	15.9	None	Good; 1 abrupt diameter change
T266	239						--		300	41.3	14.9	None	Good

* $K = \frac{P}{\ln R}$

(Table continued on next page)

NOTES

A. Tooling

1. Liner

Size: 3.050-inch in 1400-ton press, and 2.040-inch in 300-ton press (Tubes 180-182, 220, 259, 261)

Temp: 900°F.

2. Die

Type: Internal, integral, TZM insert

Temp: Ambient

B. 1200°C extrusions lubricated by means of a 16-gauge spun copper can slipped into the press liner ahead of the billet. 1300°C and 1500°C billets glass lubricated.

TABLE 4

DIMENSIONS (INCHES) OF TUNGSTEN - 25 $^{W}/o$ RHENIUM RE-EXTRUDED TUBING

Tube No.	Stock Tube					Tube					
	No.	OD		Wall		Length Evaluated	Position	OD		Wall	
		Max.	Min.	Avg.	Range			Max.	Min.	Max.	Min.
T180	174	0.373	0.372	0.040	---	47		0.086	0.081	---	---
T181		0.373	0.372	0.040	---	6		0.066		---	---
T184		0.373	0.372	0.040	---	56		0.082	0.075	---	---
T185		0.373	0.372	0.040	---	78		0.063	0.060	---	---
T182	178-2	0.378	0.375	0.041	---	49	F	0.086	0.081	0.008	0.007
T186		0.378	0.377	0.042	---	52	F	0.084	0.076	0.009	0.008
							R	---	---	0.009	0.008
T187		0.377	0.375	0.041	---	71		0.062	0.052	---	---
							F	---	---	0.0101	0.0095
T190	178-2	0.380	0.376	0.040	0.003	53	M	0.083	0.076	0.0106	0.0097
							R	---	---	0.0115	0.0083
T191		0.377	---	---	---	30	M	0.082	0.075	---	---
T192		0.379	---	---	---	--		0.070	0.066	---	---
T205	194	0.375	0.370	0.435	0.001		F	0.102	0.098	0.012	
							R	0.085	0.083		
T206		0.375	0.369	0.435	0.001		F	0.099	0.097	0.011	
							R	0.093	0.091		
T209	194	0.376	0.374	0.435	0.001						
T210		0.375	0.374	0.044	0.0015	32	F	0.100	0.098		
							R	0.100	0.097		

(Table continued on next page)

TABLE 4 (CONTINUED)

Tube No.	Stock Tube					Tube					
	No.	OD		Wall		Length Evaluated	Position	OD		Wall	
		Max.	Min.	Avg.	Range			Max.	Min.	Max.	Min.
T254		0.375	0.374	0.0415	0.001	28	F R	0.092 0.095	0.086 0.093		
T255		0.374	0.374	0.0415	0.001	24	F R	0.094 0.093	0.089 0.088		
T256		0.374	0.373	0.0415	0.001	24	F R	0.092 0.092	0.089 0.090		
T257	239	0.373	0.372	0.0415	0.001	30	F R	0.089 0.089	0.088 0.088		
T258		0.374	0.374	0.0415	0.001	26	F R	0.093 0.096	0.083 0.082		
T259		0.375	0.374	0.0375	0.001	14	F R	0.090 0.089	0.087 0.088		
T260		0.372	0.371	0.039	0.002	--		---	---		
T261		0.372	0.371	0.039	0.002	18	F R				

TABLE 4 (CONTINUED)

Tube No.	Stock Tube					Tube					
	No.	OD		Wall		Length Evaluated	Position	OD		Wall	
		Max.	Min.	Avg.	Range			Max.	Min.	Max.	Min.
T212	199	0.375	0.373	0.042	0.003	30	F R	0.098 0.097	0.097 0.095		
T213		0.373	0.370	0.042	0.001	30	F R	0.099 0.097	0.097 0.096		
T214		0.371	0.369	0.040	0.002	26	F R	0.098 0.096	0.095 0.094		
T215A		0.371	0.371	0.041	0.001						
T215B						6	F	0.072	0.069	0.009	0.007
T216		0.372	0.369	0.042	0.002	15	F R	0.127 0.128	0.125 0.126	0.0143	0.0138
T220	196	0.375	0.375	0.039	---						
T221		0.277	0.275	0.023	0.001	24	F R	0.080 0.079	0.078 0.078		
T222		0.280	0.275	0.022	0.001	30	F M R	0.078 0.075 0.077	0.077 0.074 0.076		
T223	199	0.375	0.375	0.040	0.002	22	F M R	0.100 0.097 0.098	0.097 0.095 0.097	0.0075	
T224		0.375	0.375	0.042	---	22	F M R	0.098 0.100 0.101	0.096 0.099 0.100		

(Table continued on next page)

TABLE 4 (CONTINUED)

Tube No.	Stock Tube					Tube					
	No.	OD		Wall		Length Evaluated	Position	OD		Wall	
		Max.	Min.	Avg.	Range			Max.	Min.	Max.	Min.
T225	217	0.380	0.378	0.047	0.002	30	F M R	0.101 0.105 0.106	0.099 0.103 0.103		
T232	217	0.375	0.373	0.044	---	30	F R	0.096 0.101	0.094 0.099		
T233		0.375	0.373	0.044	---	38	F R	0.090 0.095	0.088 0.094		
T234		---	0.373	0.044	---	42	F R	0.097 0.096	0.095 0.095		
T235		---	0.373	0.044	---	8	F R	0.088 0.091	0.087 0.089		
T246	217	0.372	0.371	0.044	0.002	26	F R	0.095 0.098	0.094 0.097		
	199	0.373	0.371	0.036	---	--		---	---		
	217	0.375	0.375	0.0435	0.001	--		---	---		
T247	217	0.373	0.371	0.044	0.002						
	239	0.372	0.372	0.040	0.002	24	F R	0.094 0.095	0.092 0.093		
	217	0.372	0.371	0.0435	0.003	28	F R	0.094 0.095	0.093 0.095		

(Table continued on next page)

TABLE 4 (CONTINUED)

Tube No.	Stock Tube					Tube					
	No.	OD		Wall		Length Evaluated	Position	OD		Wall	
		Max.	Min.	Avg.	Range			Max.	Min.	Max.	Min.
T248	217	0.372	0.372	0.044	0.002	14	F	0.127	0.125		
							R	0.129	0.128		
	239	0.373	0.363	0.038	0.004						
	217	0.372	0.372	0.0445	0.003	14	F	0.128	0.127		
							R	0.127	0.126		
T249	236	0.298	0.297	0.032	0.002	12	F	0.103	0.102		
							R	0.108	0.107		
	237	0.297	0.295	0.0315	0.001	14	F	0.100	0.098		
							R	0.100	0.098		
T250	217	0.373	0.372	0.045	0.002	--		---	---		
		0.373	0.373	0.045	0.002	36	F	0.100	0.099		
							R	0.100	0.096		
		0.373	0.373	0.045	0.002	28	F	0.099	0.095		
							R	0.099	0.093		
T251	217	0.370	0.368	0.0425	0.001	--		---	---		
	239	0.372	0.371	0.0385	0.001	--		---	---		
	217	0.372	0.372	0.0435	0.001	28	F	0.096	0.093		
							R	0.097	0.095		

(Table continued on next page)

TABLE 5

SUMMARY OF DRAW TESTS USING TUNGSTEN - 25 $^{W}/_{o}$ RHENIUM

Main Table	Draw Temp (°C)	Drawing Technique Used										
		Sinking	Deformable Mandrel	Moving Mandrels						Plugs		
				M-2 (R_c46)	M-2 (R_c60)	M-3 (R_c60)	H-12 (R_c50)	H-13 (R_c39)	L-718 (R_c46)	Not Identified	M-2 (R_c46)	M-3 (R_c60)	NM-100 (R_c60)
6 (0.30 x 30 as-extruded stock)	300	132A,B								132C,D,E 134A			
	425	171D,E 172E				165C	172F 187A						
	450	171A 172A 184E		172C		172B 184A,B,C,D			171C				
	510	139D 144A,B 145A,B 146B 165A,B 167A,F 168A,F 169A,F 183A				167B,C,D,E 168B,C,D,E 169B,C,D,E 186A			182A,B,C 183B,C	133A 134B,C,D,E 139A,B,C 146A	102D,E,F,G		
7 (3/8 x 30-40 stock)	250									128G			
	300			135A						128F			
	350									128E			
	400			128D									
	425										128C		
	450										128B		

(Table continued on next page)

TABLE 5 (CONTINUED)

Main Table	Draw Temp (°C)	Drawing Technique Used												
		Sinking	Deformable Mandrel	Moving Mandrels							Plugs		
				M-2 (R_c 46)	M-2 (R_c 60)	M-3 (R_c 60)	H-12 (R_c 50)	H-13 (R_c 39)	L-718 (R_c 46)	Not Identified	M-2 (R_c 46)	M-3 (R_c 60)	NM-100 (R_c 60)
7 (3/8 x 30-40 stock)	510	140A 149G 150A,B 151A,B,F 152A,B 158A,B,C,D,E 159A,B,C,D,E 160A,B 161A,B,C,D 162A,B,C 163A,B,C 164A,B 192D,E,F 193D,E,F,G 194D,E,F,G		135B,C,D,E 141A,B,C 151E 152F,G 158G 159G 160D 161E 162D 163D 164C,D,E	151G,H,I 152G 159H,I,J 162E,F 163E,F,G,H	152H,I,J		160C			102D,E,F,G 135C 138A,B,C,D,E 140B 149A,B,C,D, E,F 151C,D 152C,D,E 158F 159F 161E		
	580	192C		193A,B 194B						102J 126B,C,D,E, F,G,H,I, J,K	191A 192A,B 193C 194A,C		126A 127A 128A
8 (0.1 x 10 stock)	300	129C,D 131C,D,E											
	510	166E	153A,B,C,D,E 166A,B,C,D										
	580	124A,B,C 125A,B,C 129A,B 131A,B	123A,B,C,D 148A,B,C,D										

(Table continued on next page)

TABLE 5 (CONTINUED)

Main Table	Draw Temp (^oC)	Drawing Technique Used											
		Sinking	Deformable Mandrel	Moving Mandrel							Plugs		
				M-2 (R_c46)	M-2 (R_c60)	M-3 (R_c60)	H-12 (R_c50)	H-13 (R_c39)	L-718 (R_c46)	Not Identified	M-2 (R_c46)	M-3 (R_c60)	NM-100 (R_c60)
9 (miscellaneous sizes)	510	98E 99G,H,I,J 101G 103G,H,I,J 147C,D,E 154A,B,C,D,E,F,G,H,I,J 155A,B,C,D,E,F,G,H,I,J 156A,B,C,D,E,F,G,H,I,J 179A,B,C 180A,B,C 181A,B,C	136A 137A 142A,B 143A,B 157A 173A 175A 176A 177A 178A 190A							147A,B	98D,F 101D,E,F 103D,E,F	70C	77C
	580	99G,H,I,J 100G,H,I									99D,E,F 100D,E,F		

TABLE 6

Draw Series D	Extrusion Tube T	Drawing Reduction (% per pass)(1)											Cum. Red. (%) (2)	Passes Followed by Anneal	Remarks
		A	B	C	D	E	F	G	H	I	J	K			
132	177	0.0(3)	1.5(3)	3.0(3)	3.5(3)	[25](3)	-	-	-	-	-	-	7.9	C	Point broke in pass E.
133	195	20.1	-	-	-	-	-	-	-	-	-	-	-	-	Cracked in reeling.
134	195	[20](3)	5.1	2.7	8.9	0.0	-	-	-	-	-	-	15.8	B,C	Broke (spiral and transverse cracks) while drawing in pass A. Drew O.K. in subsequent passes.
139	195	2.8	4.1	10.0	0.0	-	-	-	-	-	-	-	22.2	B,C	Reeling difficult.
144	195	2.7	1.8	-	-	-	-	-	-	-	-	-	4.4	A	Cracked in fused NaOH before anneal after pass B.
145	193	0.9	6.5	-	-	-	-	-	-	-	-	-	6.9	A	Cracked in fused NaOH before anneal after pass B.
146	200	6.2	4.3	-	-	-	-	-	-	-	-	-	10.2	A	Cracked in banana-peel fashion 14 hours after pass B, before anneal.
165	218	4.2	3.3	33.4(3)	-	-	-	-	-	-	-	-	7.2	A,B	Cracked while drawing in pass C.
167	227	1.4	7.7	4.3	13.3	10.8	0.6	-	-	-	-	-	32.3	A,B,C,D,E,F	Reeled at 600-650°C after all but pass F (a sink).(4)
168	227	1.4	5.6	2.2	12.7	10.7	0.6	-	-	-	-	-	29.5	A,B,C,D,E,F	Reeled at 600-650°C after all but pass F (a sink); several short cracks visible.
169	227	1.4	3.4	5.3	11.9	11.4	0.6	-	-	-	-	-	30.2	A,B,D,E,F	Reeled at 600-650°C after all but pass F (a sink); several short cracks visible.(4)
171	218	2.2(3)	2.2(3)	9.2(3)	2.0(3)	1.4(3)	-	-	-	-	-	-	15.9	A,B,C,D,E	Reeled at 480-510°C after passes A,B,C; reeling not required after pass D (a sink); reeled at 425°C after pass E.

(Table continued on next page)

TABLE 6 (CONTINUED)

HISTORY OF TUBES DRAWN AT 510°C FROM .30-IN. O.D. x 30-MIL WALL STOCK

Draw Series D	Extrusion Tube T	Drawing Reduction (% per pass)(1)											Cum. Red. (%) (2)	Passes Followed by Anneal	Remarks
		A	B	C	D	E	F	G	H	I	J	K			
172	218	1.3(3)	4.7(3)	5.2(3)	10.0(3)	3.4(3)	2.9(3)	-	-	-	-	-	24.7	A,B,C,D,E,F	Reeled at 510°C after passes A,B,C,D; reeling unnecessary after pass E; reeled at 425°C after pass F.
182	227	3.1	13.7	11.5	-	-	-	-	-	-	-	-	25.9	A	Reeling at 650-675°C troublesome; couldn't remove mandrel after pass C; mandrel deformed in passes B and C.
183	227	1.8	9.8	13.3	-	-	-	-	-	-	-	-	10.8	A	Reeling at 650-675°C troublesome; couldn't remove mandrel after pass C; mandrel softened and deformed in each pass.
184	228	5.9(3)	11.6(3)	8.8(3)	8.9(3)	0.0(3)	-	-	-	-	-	-	30.9	B,C,D,E	Reeled at 480°C; shipped to P and W.
186	228	20.6	-	-	-	-	-	-	-	-	-	-	20.6	A	Ends cracked and mandrel softened and deformed in reeling at 675°C (cf Table 5)
187	226	10.4(3)	-	-	-	-	-	-	-	-	-	-	10.4	A	Mandrel softened and deformed in reeling at 480°C (cf Table 5).

(1) Attempted reduction indicated by value in brackets.

(2) Successfully achieved; may have cracked subsequently.

(3) Draw done at different temperature than 510°C (see Table 5).

(4) Same mandrel reused for passes A,B,C,D.

TABLE 7

HISTORY OF TUBES DRAWN AT 510°C FROM 3/8-IN. O.D. x 30-40-MIL WALL STOCK

Draw Series D	Extrusion Tube T	Drawing Reduction (% per pass)(1)											Cum. Red. (%) (2)	Passes Followed by Anneal	Remarks
		A	B	C	D	E	F	G	H	I	J	K			
102	168	See NMI-1264			11.5	4.1	20	[20]	[20]	-	-	-	54	None	Ductile fracture in pass G; Broke at room temperature in H.
126	178-2	5.1	15.0(3,4)	10.8(3,4)	7.9(3,4)	17.3	6.7	8.9	6.5	8.3	3.0	0.7	61.2	H	Plug necked in pass A; cracked at rear during reeling after passes B,C,D,E,G,H,I,J; sunk in passes F and K. First piece of 1/4-20 produced by moving mandrel technique.
127	179-2	[21]	-	-	-	-	-	-	-	-	-	-	-	-	NM-100 plug broke at connection to draw bench; draw no good.
128	179-2	9.3	12.3	2.6	8.5	7.8	16.0	18.0	-	-	-	-	45.1	C	Plug necked in passes A,B,C; rear of tube cracked in reeling after passes D,E,F. Tube cracked in drawing pass G for 55% cum. red.
135	194	9.9	9.5	8.0	3.1	14.1	-	-	-	-	-	-	26.2	None	Plug necked in pass C; passes A and B good moving mandrel draws; partly cracked after reeling in pass D; fully cracked at end of draw E in attempt for 37% cum. red.
138	198	3.1	4.0	11.7	6.7	3.5	-	-	-	-	-	-	22.2	C	Plug necked in pass B; cracked at point in draw D and at rear in reeling after pass D; fully cracked in reeling after pass E.
140	198	0.6	[9]	-	-	-	-	-	-	-	-	-	0.6	Tube recrystallized before pass A	Point kept breaking in pass B attempts; fractures appear to be intergranular.

(Table continued on next page)

TABLE 7 (CONTINUED)

Draw Series D	Extrusion Tube T	Drawing Reduction (% per pass)(1)											Cum. Red. (%) (2)	Passes Followed by Anneal	Remarks
		A	B	C	D	E	F	G	H	I	J	K			
141	198	[22]	5.3	5.9	-	-	-	-	-	-	-	-	11	Tube recrystallized before pass A	Point broke in pass A; broke at ends in reeling after pass B; cracked length in reeling after pass C. Fractures appear to be intergranular.
149	168	4.8	4.0	6.1	7.2	11.3	13.7	4.2	-	-	-	-	38.3	None	O.K. until last pass (G) when attempt at 41% cum. red. produced lengthwise crack.
150	197	0.6	6.1	-	-	-	-	-	-	-	-	-	6.7	A	Cracked after draw in attempt to remove soft steel tube accidentally stuck inside W-25 Re.
151	197	1.5	7.5	9.8	8.5	9.9	3.2	6.9	13.9	9.6	-	-	52.1	Each pass	Short crack in reeling after pass E. Reeled at 480°C after passes E,G,H,I.
152	197	2.0	7.4	-	8.6	8.9	12.9	3.8	8.4	13.7	9.1	-	54.5	Each pass (except C)	Point broke in pass C; reeled at 480°C after passes F,H,I,J.
158	201	0.9	1.4	3.6	1.8	2.7	3.1	3.8	-	-	-	-	15.9	Each pass	Mostly sinking to pass E. Light plug ironing poor in pass F when O.D. .313 in. and wall 30 mils.
159	201	0.4	1.0	4.1	2.2	3.1	3.1	10.0	3.1	8.6	14.4	-	37.4	Each pass	Reeled at 480°C after pass I and J; cracked after pass J.
160	202	3.7	0.4	7.1	10.0	-	-	-	-	-	-	-	20.0	Each pass	Cracked at rear and mandrel (H-13 steel) deformed during reeling after pass C; split near point during reeling after pass D.

(Table continued on next page)

TABLE 7 (CONTINUED)

Draw Series D	Extrusion Tube T	Drawing Reduction (% per pass)(1)											Cum. Red. (%) (2)	Passes Followed by Anneal	Remarks
		A	B	C	D	E	F	G	H	I	J	K			
161	202	3.1	3.3	2.3	2.3	8.5	-	-	-	-	-	-	18.2	Each pass	Ductile fracture occurred in drawn section during pass E which started as a plug draw. Pass E completed O.K. as moving mandrel draw, but tube fractured in reeling.
162	211	3.5	2.2	3.0	10.3	3.0	8.2	-	-	-	-	-	26.7	Each pass	Cracked in reeling at 480°C after pass F.
163	211	4.0	2.2	2.7	4.6	2.8	9.4	14.3	9.4	-	-	-	46.4	Each pass	Reeled O.K. at 480°C after passes F,G,H.
164	202	0.9	2.0	12.0	8.1	7.5	-	-	-	-	-	-	27.3	A,B	Drew O.K. through pass E; loosened on mandrel by ball swaging after passes C and E.
191	211	[31]	-	-	-	-	-	-	-	-	-	-	-	-	Ductile failure in attempt at 31% cum. red. during pass A.
192	211	10.5	24.2	2.8	5.0	6.3	5.6	-	-	-	-	-	44.6	B,C,D,E,F	Attained 20-mil wall in passes B,C,D,E,F; were supposed to be sinks but very light ironing may have occurred; produced ironed 1/4-20 tubing without using moving mandrel.
193	211	11.8	6.8	14.6	1.4	5.1	6.7	[16]	-	-	-	-	38.7	D,E,F	Attempt to repeat plug draw as in 192A,B bordered on tensile failure, so moving mandrel used until pass C; pass G terminated by ductile required excessive forces; should have been same as 192F.
194	211	6.1	10.4	17.1	1.4	4.7	5.7	5.6	-	-	-	-	48	E,F	Tube bent during pass G; cracked in reeling at 480°C after pass G.

(Table continued on next page)

TABLE 7 (CONTINUED)

(1) Attempted reduction indicated by value in brackets.

(2) Successfully achieved; may have cracked subsequently.

(3) Drawn at temperature other than 510°C (cf Table 5).

(4) Used same mandrel with progressively smaller dies.

TABLE 8

HISTORY OF TUBES DRAWN AT 580°C FROM NOMINAL 0.1-IN. DIA. x 10-MIL WALL STOCK

Draw Series D	Extrusion Tube T	Drawing Reduction (% per pass)(1)											Cum. Red. (%)	Passes Followed by Anneal	Remarks
		A	B	C	D	E	F	G	H	I	J	K			
124	171-1	8.1	5.7	-	-	-	-	-	-	-	-	-	13.3	A,B	Sunk bare without mandrel; O.D. decrease of about 20 mils; point broke in pass C attempt to get 103-mil O.D.
125	171-2	9.5	6.8	-	-	-	-	-	-	-	-	-	15.6	A,B	Sunk bare without mandrel; point broke in pass C attempt to get 95-mil O.D.
129	182	4.4	2.1	3.2(2)	2.8(2)	-	-	-	-	-	-	-	12.0	A,B,C,D	Sunk bare without mandrel; achieved 69-mil O.D.
131	182	4.3	3.3	3.2(2)	3.6(2)	(2)	-	-	-	-	-	-	14.9	A,B,C,D	Same as 129; attempt at 58-mil O.D. failed in pass E when points kept breaking.
123	171-2	10.0	18.6	13.2	11.4	-	-	-	-	-	-	-	48.0	None	Drawn with inner Mo extrusion filler acting as deformable mandrel; decreased O.D. from 133 mils to 96 mils.
148	216	14.9	11.5	9.9	7.3	-	-	-	-	-	-	-	37.1	A,B,C	Drawn with inner Mo extrusion filler (from which CaC_2 core had been removed) acting as deformable mandrel; decreased O.D. from 125 to 88 mils.
153	223	3.9(2)	5.3(2)	10.0(2)	2.7(2)	10.6(2)	-	-	-	-	-	-	28.8	A,B,C,D	Drawn with inner Mo plus CaC_2 core acting as deformable mandrel. Core and Mo were readily removed after pass E. O.D. decreased from 100 to 84 mils. Best technique for 80-8 tubing.
166	224	2.0(2)	10.8(2)	11.4(2)	10.6(2)	[11](2)	-	-	-	-	-	-	30.8	A,B,C,D	Repeat of 153 except removed CaC_2 before last pass E. Point broke in pass E.

(1) Attempted reduction indicated by value in brackets.

(2) Drawn at temperature other than 580°C (cf Table 5).

TABLE 9

HISTORY OF TUBES DRAWN AT 510°C FOR SPECIAL PURPOSES

Draw Series D	Extrusion Tube T	Drawing Reduction (% per pass)[1]											Cum. Red. (%) (2)	Passes Followed by Anneal	Remarks
		A	B	C	D	E	F	G	H	I	J	K			
A. Irradiation Material (0.255-in. O.D. x 30-mil wall)															
98	169	See NMI-1264			11.4	2.5	[20]	-	-	-	-	-	28.3	None	Mandrel didn't grab in pass E; tube cracked at rear; ductile fracture at point in pass F when O.D. 0.325 in. and wall 31 mils.
99	169	See NMI-1264			10.3 (3)	6.9 (3)	20.2 (3)	5.6 (3)	8.3 (3)	3.7 (3)	2.6 (3)	-	55.4	I	High forces in pass D caused point failure several times before draw successful; tube cracked at rear in pass E; plug necked in pass F; sunk about 55 mils after pass F.
100	167	See NMI-1264			10.6 (3)	3.0 (3)	19.5 (3)	4.8 (3)	4.4 (3)	5.1 (3)	-	-	41.7	None	Plug necked in pass F; tube cracked in attempt for 45% cum. red. in pass I, the third sink pass after ironing completed in pass F.
101	167	See NMI-1264			10.0	10.6	17.2	5.3	-	-	-	-	37.7	-	Tube cracked in first sinking pass (G) for attempted cum. red. of 41% following extensive ironing.
103	168	See NMI-1264			11.6	4.5	18.6	5.1	4.8	5.1	8.9	-	53.2	F,G,H,I,J	Successfully sunk using 1200°C - 1/2 hour anneal after last ironing pass (F) and subsequent sinking passes.
147	200	12.0	10.6	3.3	2.5	2.0	-	-	-	-	-	-	27.4	B,C,D,E	Cracked at ends in reeling after passes A and B; subsequently sunk O.K. using 1400°C - 1/2 hour anneal.
154	202	2.5	3.6	1.8	2.9	3.0	3.5	2.1	4.6	3.3	5.4	-	28.5	Each pass	Decreased O.D. from 0.370 in. to 0.255 in. without difficulty using sinking for all passes and 1400°C - 1/2 hour anneal.
155	201	0.4	1.7	3.4	2.0	2.8	3.2	3.7	2.3	4.5	9.2	5.5	30.3	Each pass	Decreased O.D. from 0.370 in. to 0.255 in. without difficulty using sinking for all passes and 1400°C - 1/2 hour anneal.

(Table continued on next page)

TABLE 9 (CONTINUED)

Draw Series D	Extru-sion Tube T	Drawing Reduction (% per pass)(1)											Cum. Red. (%) (2)	Passes Followed by Anneal	Remarks
		A	B	C	D	E	F	G	H	I	J	K			
156	211	4.2	2.2	2.7	3.1	2.2	4.0	3.0	5.6	-	-	-	21.5	Each pass	Decreased O.D. from 0.355 in. to 0.255 in. using sinking for all passes and 1400°C - 1/2 hour anneals.
179	230	2.1	4.0	4.7	-	-	-	-	-	-	-	-	10.3	Each pass	Decreased O.D. from 0.300 in. to 0.255 in. using sinking for all passes and 1400°C - 1/2 hour anneals.
180	230	2.5	4.2	3.4	-	-	-	-	-	-	-	-	9.8	Each pass	Decreased O.D. from 0.300 in. to 0.255 in. using sinking for all passes and 1400°C - 1/2 hour anneals.
181	230	2.1	4.1	3.2	-	-	-	-	-	-	-	-	9.1	Each pass	Decreased O.D. from 0.300 in. to 0.255 in. using sinking for all passes and 1400°C - 1/2 hour anneals.

(Table continued on next page)

TABLE 9 (CONTINUED)

Draw Series D	Extru-sion Tube T	Drawing Reduction (% per pass)(1)											Cum. Red. (%) (2)	Passes Followed by Anneal	O.D. (in.)		Remarks
		A	B	C	D	E	F	G	H	I	J	K			Start	Finish	
B. Re-extrusion Material (drawn with inner extrusion filler as deformable mandrel)																	
136	194	1.3	-	-	-	-	-	-	-	-	-	-	1.3	A	.385	.375	O.K.
137	199	2.1	-	-	-	-	-	-	-	-	-	-	2.1	A	.390	.375	Ovality reduced from 42 to 5 mils.
142	199	0.8	1.1	-	-	-	-	-	-	-	-	-	1.8	B	.385	.375	O.K.
143	200	0.5	2.5	-	-	-	-	-	-	-	-	-	2.9	-	.320	.312	Ovality reduced from 32 to 1 mil.
157	217	2.3	-	-	-	-	-	-	-	-	-	-	2.3	A	.385	.375	O.K.
173	239	2.2	-	-	-	-	-	-	-	-	-	-	2.2	A	.380	.370	O.K.
175	217	2.3	-	-	-	-	-	-	-	-	-	-	2.3	A	.385	.376	O.K.
176	217	4.1	-	-	-	-	-	-	-	-	-	-	4.1	A	.390	.375	O.K.
177	236	2.7	-	-	-	-	-	-	-	-	-	-	2.7	A	.310	.302	O.K.
178	237	3.4	-	-	-	-	-	-	-	-	-	-	3.4	A	.310	.302	O.K.
190	239	5.5	-	-	-	-	-	-	-	-	-	-	5.5	A	.390	.375	O.K.
C. Miscellaneous																	
															Remarks		
70	117	See NMI-1264		6.6	-	-	-	-	-	-	-	-	52.2	-	M-3 (R_c60) mandrel used successfully as plug.		
77C	129	See NMI-1264		[12]	-	-	-	-	-	-	-	-	-	-	Ductile fracture of tube in drawn section; tube was 1800°C extrusion; O.D. 0.345 in., wall 32 mils at fracture. NM-100 plug looks promising.		

(1) Attempted reduction indicated by value in brackets.

(2) Successfully achieved; may have cracked subsequently.

(3) Drawn to temperature other than 510°C (cf Table 5).

TABLE 10

TESTS TO ESTABLISH CONDITIONS FOR REELING NOMINAL 1/4-20 W - 25 Re TUBING

(Roll axes inclined 144° to each other)

Mandrel Material	Draw Test	Draw Temp (°C)	Draw Reduction (%)	Reel Temp (°C)	Type Heat	Reel Passes	Effect on Tube	Effect on Mandrel
M-2 Steel R_c 48	168E			590	Torch	12	Barely loosened.	Scored; diamater decreased 1 mil.
	172C	460	5.2	510	Furnace	5	Loosened.	O.K.
	183C	510	13.3	620-680	Furnace	1	Barely loosened but stuck on mandrel.	(1)
	193A	580	11.8	450	Furnace	10	Loosened.	O.K. (2)
H-12 Steel R_c 50	171E	425	1.4	425	Furnace	15	Loosened. (3)	Softened to about R_c 48.
	172F	425	2.9	425	Furnace	5	Loosened. (3)	Softened to about R_c 47.
	187A	425	10.4	480	Furnace	6	Loosened.	Softened to R_c 44; diameter decreased 1 mil.
H-13 Steel R_c 41	160C	510	7.1	600	Torch	16	Tight.	Softened to R_c 37; diameter decreased 3 mils
Lescalloy-718 R_c 46	171C	450	9.2	480	Furnace	12	Barely loose.	Scored, bent; diameter decreased 1/2 mil. Softened to R_c 42.
	182A	510	3.1	670-700	Torch	12	Loosened. (3)	O.K.
	B	510	13.7	670	Torch	4	Loosened.	Dia. necked 1 mil.
	C	510	11.5	620-670	Furnace	3	Tight.	Dia. decreased 3 to 4 mils.

(Table continued on next page).

TABLE 10 (CONTINUED)

Mandrel Material	Draw Test	Draw Temp (^oC)	Draw Reduction (%)	Reel Temp (^oC)	Type Heat	Reel Passes	Effect on Tube	Effect on Mandrel
Lescalloy-718 R_c 46	183A	510	1.8	670-700	Torch	4	Loosened. (3)	O.K.
	B	510	9.8	670-700	Torch	3	Tight.	Softened to R_c 41; dia. decreased 3 mils

(1) Tube would not pass through reeling machine after first pass. Mandrel was stuck inside so could inspect. Shattered tube in attempt to loosen by light swaging at 900^oF which apparently defoemed mandrel.

(2) This mandrel for 193A was unusually hard - R_c 55 - and not representative of the usual M-2 mandrels used in this program.

(3) Tubes in these tests were probably barely seated on the mandrels after drawing.

TABLE 11

FABRICATION HISTORY OF W - 25 Re TUBING (1/4-inch OD x 20-MIL WALL) PRODUCED IN PILOT PRODUCTION RUN

Ident.	Length Shipped, in.[1]	Source		Drawing Reduction (%)					Passes Followed by anneal [3]	Dimensions, as Shipped (mils)		
		Ext.	Location (in.)	Pass[2] A	Pass[2] B	Pass[2] C	Pass[2] D	Cum.		OD	ID[4]	Wall[4]
D184	10.1	T228	70-80	5.9	11.6	8.8	8.9	30.9	BCDE	250	210	19-21
D185	9.8	T228	81-91	18.8	8.3	9.2	0.5	30.2	CD	250	210	20
D188	10.5	T226	78-89	12.6	8.7	8.1	0.6	27.1	CD	250	210	20
D189	8.9	T226	90-100	13.1	8.9	8.3	0.0	27.4	CD	250	210	20
D192[2]	11.6	T211	15-25	10.5	24.2	2.8	5.0	44.6	CDEF	250	210	20
D196	10.1	T229	4-16	6.2	6.9	8.2	0.0	19.9	CD	251	211	20
D197	7.2	T229	16-28	6.8	8.4	7.1	0.0	20.5	CD	250	210	20
D198	7.8	T229	28-40	7.2	8.2	7.8	0.8	22.1	CD	250	210	20
D203	13.3	T228	16-28	19.5	9.6	7.5	0.0	32.7	All Passes	250	210	19-21
D207	7.1	T230	94-105	16.1	7.4	8.6	0.5	29.4	D	251	210	21
D210	10.1	T243	13-24	5.9	5.7	9.3	0.6	20.0	D	251	210	20
D211	10.3	T243	24-34	15.3	5.0	9.1	0.0	26.9	D	250	210	20
D212	11.6	T242	3-15	7.9	6.3	9.8	0.5	22.6	BCD	250	210	20
D214	11.7	T242	27-39	6.8	6.4	8.0	0.0	19.8	BCD	250	209-210	21
D215	8.5	T244[5]	24-34	1.6	3.9	4.3	0.0	8.1	BCD	250	210-211	19
D216	9.3	T244[5]	3-13	2.2	5.0	7.8	0.0	14.4		250	211	19-21
D217	9.4	T244[5]	13-24	3.0	3.7	7.8	0.6	14.4		250	210	20-21
D218	26.4	T244[5]	34-59	1.1	9.6	6.6	0.0	16.5		249-250	210	20
D219	9.4	T244[5]	59-70	0.6	3.2	6.7	0.6	10.7		250	210	19-20
D220	33.5	T242	75-105	11.1	9.1	7.5	0.2[3]	25.6		250	210	19-20
D221	17.2	T243	34-66	5.7	9.2	9.9	0.0	23.0	All	250	210	19-20
D222	13.1	T230	70-82	15.2	8.4	7.4	0.0	28.1	Passes	250	210	19-20
D223	13.2	T228	4-16	18.5	16.3	6.9	0.0	36.5		250	210	19-20
D224	33.9	T242	39-70	10.9	11.1	7.6	0.0	26.8		248-251	210-211	19 1/2-21
D225	33.8	T244[5]	70-102	1.7	3.5	6.6	0.0	11.4		249-250	210	19-20
D226	35.1	T245	8-39	16.1	11.1	7.2	0.0	30.8		249-250	210	19-20
D227	35.1	T245	80-112	17.9	10.6	7.0	0.05	32.1		249-250	210	19-20
D228	29.0	T245	39-66	13.7	11.7	9.2	0.2	30.9		250-251	210	20

(Table continued on next page).

TABLE 11 (CONTINUED)

Ident.	Length Shipped, in.(1)	Source		Drawing Reduction (%)					Passes Followed by Anneal(3)	Dimensions, as Shipped (mils)		
		Ext.	Location (in.)	Pass(2) A	B	C	D	Cum.		OD	ID(4)	Wall(4)
D229	7.3	T207	6-14	13.8	9.9	8.4	0.0	28.8		250-251	210-211	20-21
D230(3)	30.4	T207	14-44	14.1	9.7	7.7	0.0	28.4		250-251	210	20
D231(3)	35.1	T218	69-98	2.5	5.4	6.3	0.0	13.6		250-251	210	19-21
D232	33.9	T227	4-36	13.3	9.1	7.1	0.0	26.8		250-251	210	20-21
D233	33.3	T229	72-106	14.5	9.9	7.4	0.2	28.8		250-251	210	20
D235	27.0	T243	84-111	5.8	8.4	6.8	0.0	19.6		250-251	210-211	19-21
Total	51.2											

(1) Measured to 1/16-inch, listed as decimal.

(2) Except for D192, A, B and C are ironing passes with a moving mandrel; D is a sinking pass. For D192, A and B are ironing passes with a stationary mandrel (plug draw) reducing the wall thickness to 20 mils; all the subsequent draws are considered sinks (no wall reduction) but plugs were used in C, D and F as a precaution to restrict decrease in ID. Reductions in E and F, 6.3 and 5.6 percent.

(3) Half hour at 1400°C in wet A-15H_2; generally preceded by vapor blast on outside; lengths greater than 18 inches annealed "end for end", i.e. each end annealed separately in furnace.

(4) Measured only at ends.

(5) Thickness of sleeve (and extruded tube) somewhat lower than others.

(6) Draw 220D received "end for end" anneal for half hour at 1300°C before 1400°C anneal.

TABLE 12

DATA SUMMARY FOR TUNGSTEN - 25 $^{w}/_{o}$ RHENIUM EXTRUSION SLEEVES

Sleeve No.	Source Ident.	Re Content (1) $^{w}/_{o}$	Nominal Dimensions (in.)		Used for NMI Billet No.	Room Temp. (2) DPH (kg/mm^2)	Density (3)	
			Diam.	Wall			gm/cc	$^{o}/_{o}$ T.D.
A. Vendor A Sleeves								
190	--	24.9	1.5	.04	--	382	19.02	97.0
192	--	25.0	1.0	.18	188	425	19.20	98.0
194	--	25.2	1.8	.15	202	405	18.95	96.6
196	--	25.1	1.5	.16	193	397	18.95	96.6
197	--	25.0	1.5	.16	194	411	18.95	96.6
198	--	25.0	1.5	.16	198	412	19.07	97.3
199	--	25.1	1.5	.16	199	420	19.02	97.0
200	--	24.1	1.5	.16	200	405	18.98	96.9
208	--	25.0	1.1	.08	195	422	19.29	98.4
209	--	25.2	1.1	.08	196	420	19.33	98.6
210	--	25.1	1.1	.10	207	393	18.87	96.3
223	--	25.0	1.5	.16	236	379	18.81	96.0
224	--	24.9	1.5	.16	237	402	18.97	96.7
225	--	25.0	1.5	.16	238	409	18.65	95.2
226	--	25.0	1.5	.16	239	405	19.02	97.1
227	--	25.0	1.5	.16	240	406	19.20	98.0
228	--	24.9	1.5	.16	241	393	19.10	97.5

(Table continued on next page.)

TABLE 12 (CONTINUED)

Sleeve No.	Source Ident.	Re Content[(1)] w/o	Nominal Dimensions (in.) Diam.	Nominal Dimensions (in.) Wall	Used for NMI Billet No.	Room Temp. DPH[(2)] (kg/mm^2)	Density[(3)] gm/cc	Density[(3)] o/o T.D.	ASTM Grain Size No.
B. Bureau of Mines Sleeves									
211	T-97	25.0	1.4	.11	211	435	19.18	97.7	6-7
212	T-124	24.1	1.5	.17	217	411	19.21	98.0	6-7
213	T-117	24.8	1.1	.10	218	412	19.15	97.6	6-7
214	T-115	24.9	1.1	.10	226	399	19.08	97.4	6-7
215	T-118	25.0	1.1	.10	227	406	18.99	96.9	6-7
216	T-113	24.8	1.1	.10	228	415	19.19	97.8	6-7
217	T-114	24.8	1.1	.10	229	454	19.28	98.4	6-7
218	T-119	25.0	1.1	.10	230	403	19.27	98.4	6
219	T-111	24.9	1.1	.09	242	415	19.23	98.1	
220	T-112	24.6	1.1	.10	243	421	19.22	98.1	
221	T-125	25.1	1.1	.09	244	377	18.90	96.5	
222	T-126	25.0	1.1	.10	245	405	18.89	96.4	
229	T-127	25.1	1.1	.10		409	19.44	99.2	
230	T-133	25.0	1.1	.10		414	19.34	98.6	
231	T-134	24.6	1.1	.10		412	19.25	98.3	
232	T-128	25.3	1.1	.10		408	19.11	97.6	
233	T-132	24.8	1.1	.10		423	19.32	98.6	
234	T-138	25.0	1.1	.10		430	19.16	97.8	
235	T-139	24.9	1.1	.10		412	19.08	97.4	
236	T-140	24.7	1.5	.15		411	18.86	96.3	
237	T-141	25.0	1.5	.13		403	18.87	96.3	
238	T-130	24.8	1.2	.12		420	19.32	98.6	7
239	T-131	24.7	1.2	.16		426	19.32	98.6	6-7

(Table continued on next page.)

TABLE 12 (CONTINUED)

Sleeve No.	Source Ident.	Re Content[1] w/o	Nominal Dimensions (in.)		Used for NMI Billet No.	Room Temp. DPH[2] (kg/mm^2)	Density[3]		ASTM Grain Size No.
			Diam.	Wall			gm/cc	o/o T.D.	
B. Bureau of Mines Sleeves									
240	T-135	24.8	1.2	.13		420	19.31	98.5	6-7
241	T-142	25.1	1.1	.10		402	19.23	98.2	6
242	T-144	25.1	1.1	.10		395	19.06	97.3	6-7
243	T-145	24.8	1.1	.10		412	19.11	97.6	7
244	T-146	25.0	1.1	.10		410	19.08	97.4	6-7
245	T-156	25.1	1.1	.10		395	18.76	95.7	7
246	T-157	25.1	1.2	.14		440	19.24	98.2	7
247	T-162	25.1	1.1	.10		413	19.04	97.2	7
248	T-163	25.0	1.1	.10		410	18.95	96.6	7
249	T-164	25.2	1.1	.10		400	19.06	97.3	7

(1) Analysis supplied by source.

(2) DPH measured with 2.5 kg load; average of 5 impressions.

(3) Determined by Archimedes method; theoretical density taken as 19.6 gm/cm^3.

TABLE 13

RING COMPRESSION TEST RESULTS FOR PRIMARY EXTRUDED TUBES 188, 207 AND 211

Condition	Position	Spec. No.	Dimensions(1) (in.) OD	Wall	Test Temp. (°C)	Deflection (in.)	Percent of Max. Defl.(2)	Load (lb.) at 0.025" Deflection Total	Per inch of length
A. Tube 188									
As-Ext.	Front	1	.254	.022	300	.021	10.0	ND	ND
		2	.254	.022	400	.026	12.4	124	599
		3	.255	.022	500	.046	22.0	114	573
		4	.254	.022	600	.072	34.4	112	538
		5	.255	.022	700	.132	62.9	109	548
Vac. Annealed 1 Hr. 1800°C.	Front	6	.253	.022	300	.112	54.1	102	504
		7	.254	.022	400	.129	61.7	96	459
		8	.253	.022	500	.092	44	85	424
		9	.254	.022	600	.095	45	86	406
		10	.254	.022	700	.184	88.1	84	408
B. Tube 207									
As-Ext.	Front	1	.286	.026	400	.018	7.7	ND	ND
		2	.285	.027	500	.022	9.5	ND	ND
		3	.286	.026	600	.040	17.1	118	735
		4	.284	.024	700	.045	19.1	122	658
	Rear	5	.292	.026	400	.020	8.4	ND	ND
		6	.291	.024	500	.032	13.2	94	604
		7	.296	.026	600	.040	16.5	121	730
		8	.295	.026	700	.050	20.6	110	681
Vac. Annealed 1 Hr. 1800°C.	Front	11	.286	.026	300	.111	47.6	125	734
		9	.286	.026	400	.126	54.1	110	653
		10	.286	.026	500	.140	60.1	108	607
		12	.286	.026	600	.118	50.6	90	584
	Rear	16	.294	.026	300	.131	54.3	120	686
		13	.296	.026	400	.144	59.2	102	650
		14	.294	.026	500	.126	52.2	102	607
		15	.296	.026	600	.137	56.3	106	579

(Table continued on next page)

TABLE 13 (CONTINUED)

Condition	Position	Spec. No.	Dimensions(1) (in.) OD	Wall	Test Temp. (°C)	Deflection (in.)	Percent of Max. Defl.(2)	Load (lb.) at 0.025" Deflection Total	Per inch of length
C. Tube 211									
As Ext.	Front	1	.363	.032	400	.029	9.7	176	900
		2	.361	.032	500	.072	24.1	167	826
		3	.358	.030	600	.095	31.7	158	796
		4	.364	.030	700	.089	29.2	148	731
	Center	5	.356	.030	400	.060	20.2	146	743
		6	.355	.030	500	.129	43.6	143	686
		7	.356	.030	600	.156	52.5	137	698
		8	.356	.030	700	.076	32.3	143	683
	Rear	9	.360	.028	400	.056	18.5	137	728
		10	.361	.030	500	.121	40.0	150	769
		11	.357	.028	600	.140	46.6	141	678
		12	.358	.028	700	.123	40.8	140	695
Vac Annealed 1 Hr. 1800°C.	Front	15	.361	.030	300	.123	41.0	142	698
		13	.360	.031	400	.154	51.6	140	682
		14	.362	.032	500	.173	57.8	128	623
		16	.363	.032	600	.144	48.0	122	603
	Center	19	.358	.032	300	.125	42.4	148	700
		17	.360	.032	400	.160	53.8	130	654
		18	.358	.032	500	.176	59.6	126	615
		20	.359	.032	600	.085	28.7	119	572
	Rear	23	.360	.030	300	.119	39.8	138	683
		21	.362	.030	400	.154	51.2	132	657
		22	.362	.032	500	.160	53.5	118	603
		24	.361	.030	600	.101	33.7	107	559

(1) Average of maximum and minimum.

(2) Percent of maximum deflection is defined as the deflection x 100/maximum possible deflection where the maximum possible deflection = OD - 2x Wall = ID.

ND = not determined.

TABLE 14

RING COMPRESSION RESULTS FOR TUBES 178-2 AND 179-2
BEFORE AND AFTER ANNEALING

Condition[1]	Spec. No.	Dimensions[2] (in.) OD	Wall	Test Temp. (°C)	Deflection (in.)	Percent of Max. Defl.[3]	Load (lb.) at 0.025" Deflection Total	Per inch of length
A. Tube 178-2; Front Position								
As Extruded	11	.377	.042	400	.053	18.1	355	1720
	12	.378	.042	600	.116	39.6	320	1600
1 Hr. - 1000°C	1	.376	.042	400	.138	47.2	361	1720
	2	.377	.042	600	.149	51.0	328	1575
1 Hr. - 1200°C	4	.376	.042	400	.125	42.7	347	1720
	3	.378	.042	600	.162	54.9	304	1550
1 Hr. - 1400°C	6	.378	.042	400	.142	48.5	333	1630
	5	.376	.042	600	.181	61.7	296	1490
1 Hr. - 1600°C	7	.374	.042	400	.189	65.0	287	1435
	8	.378	.042	600	.172	58.6	250	1330
1 Hr. - 1800°C	9	.376	.042	400	.150	51.3	256	1315
	10	.378	.042	600	.112	38.2	232	1125
B. Tube 179-2; Front Position								
As Extruded	11	.378	.042	400	.126	42.9	328	1690
	12	.377	.042	600	.114	39.1	309	1540
1 Hr. - 1000°C	1	.376	.043	400	.145	50.0	323	1615
	2	.376	.042	600	.127	43.6	304	1525
1 Hr. - 1200°C	4	.378	.044	400	.147	50.5	345	1800
	3	.378	.044	600	.140	48.2	334	1600
1 Hr. - 1400°C	5	.377	.043	400	.105	36.1	330	1590
	6	.378	.044	600	.162	55.6	322	1600
1 Hr. - 1600°C	7	.378	.042	400	.154	52.2	286	1410
	8	.379	.044	600	.151	51.9	293	1440
1 Hr. - 1800°C	9	.379	.044	400	.098	33.6	298	1440
	10	.378	.042	600	.078	26.5	233	1080

(1) Time and temperature are conditions used for vacuum annealing.
(2) Average of maximum and minimum.
(3) Percent of maximum deflection is defined as the deflection x 100/maximum possible deflection where the maximum possible deflection = OD - 2 x Wall = ID.

TABLE 15

RING COMPRESSION RESULTS FOR TUBES 198 AND 199
BEFORE AND AFTER ANNEALING

Heat Treatment(1) Temp. (°C)	Heat Treatment(1) Time (min)	Spec. No.	Dimensions(2) (in.) OD	Dimensions(2) (in.) Wall	Test Temp. (°C)	Deflec-tion (in.)	Percent of Max. Defl.(3)	Load (lb.) at 0.025" Deflection Total	Load (lb.) at 0.025" Deflection Per inch of length
A. Tube 198									
1800	15	1	.378	.044	400	.060	20.2	305	1486
		2	.378	.044	500	.054	18.6	322	1380
		4	.378	.044	600	.052	17.9	254	1280
		3	.378	.044	700	.052	18.0	257	1170
B. Tube 199									
As Extruded		2	.378	.044	400	.019	6.6	ND	ND
		1	.378	.044	500	.024	8.3	ND	ND
1700	15	10	.378	.043	400	.114	39.0	298	1550
		9	.376	.044	500	.122	42.1	284	1450
	30	12	.377	.043	400	.115	39.5	283	1490
		11	.378	.043	500	.069	23.6	278	1400
	60	14	.376	.043	400	.144	49.7	280	1480
		13	.378	.043	500	.124	42.5	296	1370
1800	15	4	.378	.044	400	.112	38.5	286	1460
		3	.378	.043	500	.106	36.3	252	1305
	30	6	.378	.044	400	.058	20.0	298	1475
		5	.378	.044	500	.133	45.9	252	1340
	60	8	.378	.043	400	.110	37.7	307	1410
		7	.378	.043	500	.145	49.6	260	1340

(1) Tube 198 annealed in A-15 H_2; self-resistance heated.
Tube 199 annealed in vacuum.

(2) Average of maximum and minimum.

(3) Percent of maximum deflection is defined as the deflection x 100/maximum possible deflection where the maximum possible deflection = OD - 2 x Wall = ID.

ND = not determined.

TABLE 16

SUMMARY OF RING COMPRESSION BEHAVIOR FOR PRIMARY EXTRUDED TUNGSTEN-RHENIUM TUBING

Tube No.	Position	RCTT(°C)(1)		
		As-Ext.	Annealed 1 Hr. 1400°C	Annealed 1 Hr. 1800°
177(2)	Front Rear	710 700	460 450	170 230
188	Front	660		(> 300)
207	Front Rear	> 700 > 700		(340) > 300
211	Front Center Rear	> 700 (580) > 700		(380) (360) (390)

(1) RCTT is the temperature for failure at 50 percent of maximum deflection (see footnote 2, Table 13). Uncertain values enclosed in parentheses.

(2) Earlier data (see NMI-1264 Table 42) No graphite core.

TABLE 17

RING COMPRESSION RESULTS FOR TUBING RE-EXTRUDED WITH ALL-MOLYBDENUM CORE

Condition	Position	Spec. No.	Dimensions(1) (in.) OD	Dimensions(1) (in.) Wall	Test Temp. (°C)	Deflection (in.)	Percent of Max Defl.(2)
A. Tube 171							
As-Extruded	Front	1	.130	.014	400	.012	11.7
		2	.130	.014	500	.017	16.5
		3	.132	.014	600	.018	17.2
		4	.130	.014	700	.010	9.7
B. Tube 182							
As-Extruded	Front	1	.082	.006	400	.007	10.0
		2	.080	.006	500	.008	11.4
		3	.082	.006	600	.008	11.4
		4	.082	.006	700	.001	15.7
Annealed 1 Hr. - 1000°C	Front	16	.082	.006	400	.006	8.5
		17	.082	.006	500	.007	9.7
		18	.082	.006	600	.008	11.4
		19	.082	.006	700	.014	19.4
Annealed 1 Hr. - 1400°C	Front	9	.082	.006	200	>.048	>68.5
		8	.082	.006	300	>.046	>65.6
		6	.082	.006	400	>.048	>68.5
		7	.084	.006	500	>.048	>66.6
Annealed 1 Hr. - 1600°C	Front	11	.082	.006	300	>.040	>57.1
		10	.082	.006	400	>.046	>65.6
Annealed 1 Hr. - 1800°C	Front	15	.082	.006	200	>.046	>65.6
		14	.082	.006	300	>.052	>74.3
		12	.082	.006	400	>.050	>71.4
		13	.082	.006	500	>.048	>68.5

(Table continued on next page)

TABLE 17 (CONTINUED)

Condition	Position	Spec. No.	Dimensions(1) (in.) OD	Dimensions(1) (in.) Wall	Test Temp. (°C)	Deflection (in.)	Percent of Max Defl.(2)
C. Tube 186							
As-Extruded	Front	1	.078	.006	300	.006	9.1
		2	.079	.006	400	.009	13.4
		3	.078	.006	500	.004	6.1
		4	.078	.006	600	.007	10.6
As-Extruded	Rear	13	.078	.006	300	.005	7.6
		14	.079	.006	400	.007	10.4
		15	.078	.006	500	.006	9.1
		16	.078	.006	600	.011	16.7
Annealed 1 Hr. - 1400°C	Front	7	.076	.006	200	.023	36.0
		8	.077	.006	250	> .050	> 77.0
		5	.076	.006	300	> .043	> 67.2
		6	.077	.006	400	> .048	> 73.8
Annealed 1 Hr. - 1400°C	Rear	19	.079	.006	200	.031	46.3
		20	.079	.006	250	> .044	> 65.7
		17	.078	.006	300	.046	71.9
		18	.079	.006	400	> .049	> 73.0
Annealed 1 Hr. - 1800°C	Front	11	.077	.006	200	.035	53.8
		12	.077	.006	250	> .050	> 76.9
		9	.077	.006	300	> .048	> 73.9
		10	.077	.006	400	> .045	> 69.2
Annealed 1 Hr. - 1800°C	Rear	23	.079	.006	200	.023	34.3
		24	.080	.006	250	> .052	> 76.4
		21	.079	.006	300	> .058	> 86.6
		22	.079	.006	400	> .047	> 70.1

(1) Average of maximum and minimum.

(2) Percent of maximum deflection is defined as the deflection x 100/maximum possible deflection, where the maximum possible deflection = OD - 2 x wall = ID.
> indicates specimen did not fracture.
Test is terminated when applied load reaches about 600 pounds.

TABLE 18

RING COMPRESSION RESULTS FOR TUBING RE-EXTRUDED WITH $Mo-CaC_2$ CORE

Position	Spec. No.	Dimensions(1) (in.) OD	Dimensions(1) (in.) Wall	Test Temp. (°C)	Deflection (in.)	Percent of Max. Defl.(2)	Max. Load (lb.) Total	Max. Load (lb.) Per inch of length
A. Tube 223; As-Extruded Condition								
Center	1	0.096	0.010	400	0.007	9.2	93	496
	2	0.096	0.010	500	0.007	9.2	91	492
	3	0.096	0.010	600	0.007	9.2	100	495
	4	0.096	0.010	700	0.007	9.2	96	486
B. Tube 224; As-Extruded Condition								
Center	1	0.101	0.011	400	0.006	7.6	110	550
	2	0.101	0.011	500	0.009	11.4	101	522
	3	0.101	0.011	600	0.012	15.2	88	508
	4	0.099	0.011	700	0.010	13.0	102	481

(1) Average of maximum and minimum.

(2) Percent of maximum deflection is defined as the deflection x 100/maximum possible deflection where the maximum possible deflection = OD - 2 x Wall = ID.

TABLE 19

HARDNESSES OF RE-EXTRUSION STOCK AND TUBING

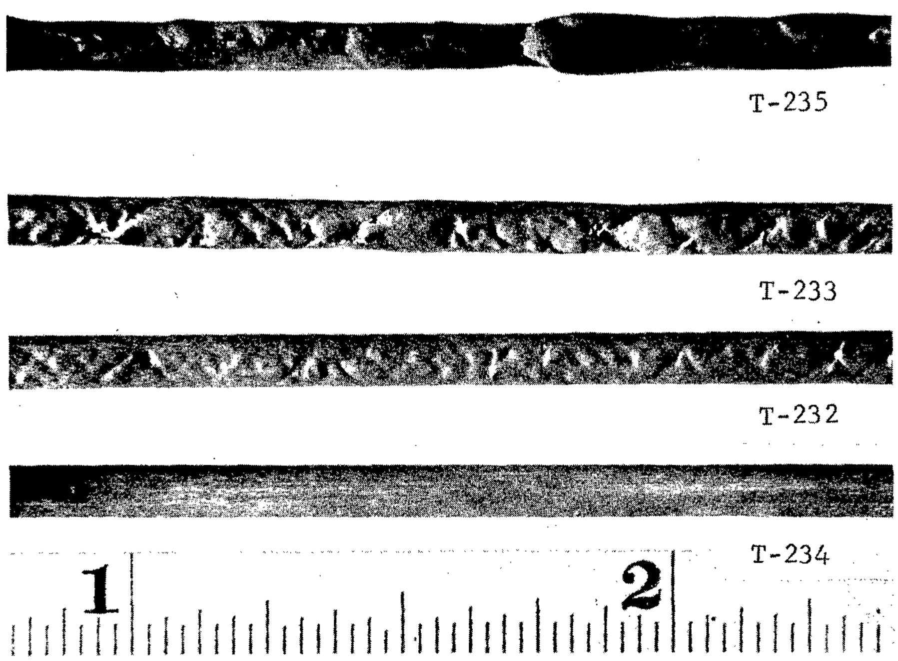
Stock			Re-Extrusion				
Identity(1)	Condition	DPH(2) (kg/mm^2)	Tube No.	Extrusion Red'n.	Extrusion Core	DPH(3) (kg/mm^2)	Tube Quality(4)
1200°C Re-Extrusions							
T 178-2	As-Ext.	529-545	182 186 190	25 25 20	Mo Mo Tamped CaC_2	743 704-698 699-705	S S S
D 137 A	As Drawn	561	205 206	19 16	Pre-Ext. CaC_2	678 677	U U
D 142 B	1/2 Hr.1350°C	538	213	16		677	U
	1 Hr.1400°C	527	214	16		659	U
	As Drawn	538	216	9		660	U
D 157 A	1 Hr.1800°C	447	232 233	16 19.5		640 634-648	U U
1500°C Re-Extrusions							
D 142 B	As Drawn	538	215B	16	Pre-Ext. CaC_2	720	(S)
D 137 A	1 Hr.1800°C	452	224	16		652	S
D 157 A	1 Hr.1800°C	447	234 235	16 19.5		570 576	U U

(1) T 178-2 = Sleeve 195
D 137 A = Tube 199 = Sleeve 199
D 142 B = Tube 199 = Sleeve 199
D 157 A = Tube 217 = Sleeve 212

(2) Average of 5 impressions made with 2.5 kg load.

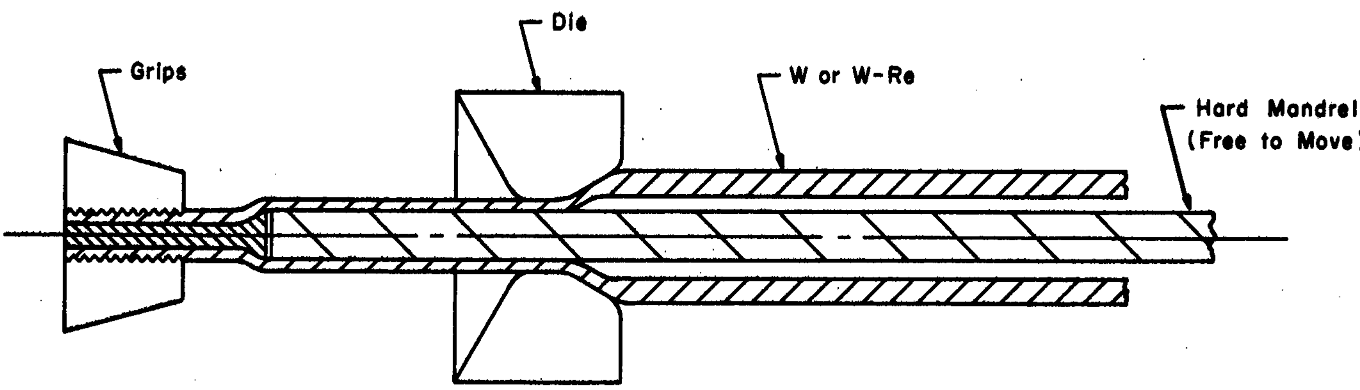
(3) Average of 5 impressions made with 500 gram load.

(4) Tube quality based on visual examinations; S indicates sound tube; U indicates tube contained chevron tears or other defects.



RF9801

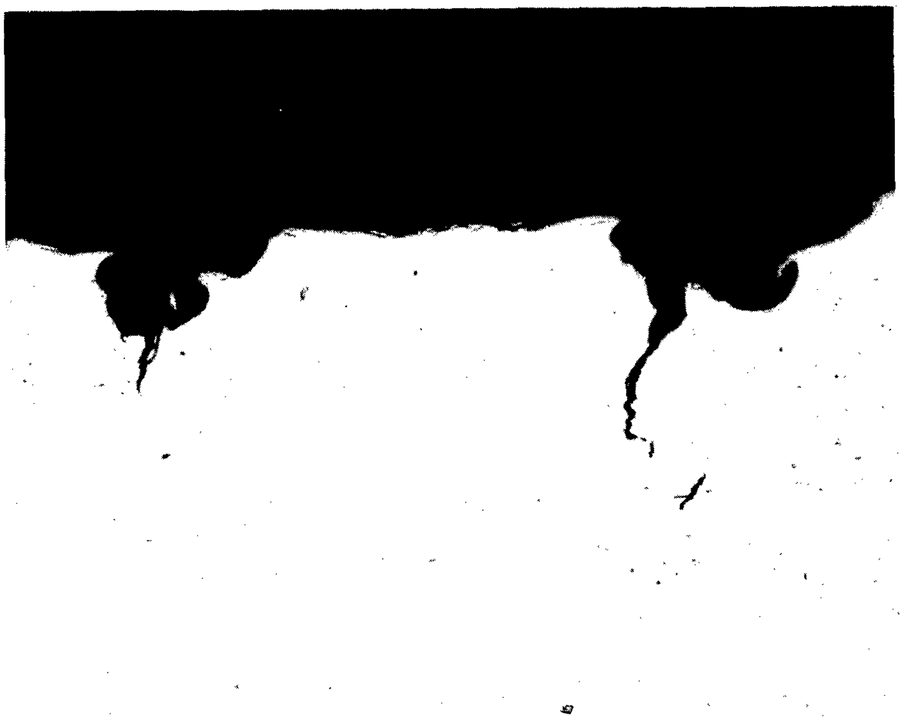
Figure 1. Four W - 25 $^{w}/o$ Re Re-Extruded Tubes Displaying Chevron Tears of Varied Severity. (The Illustrated Section of Tube 234 Contains No Tears.)



B.

Figure 2. Moving-Mandrel Technique Used to Make 1/4-Inch OD x 20-Mil Wall W - 25 $^{w}/_{o}$ Re Tubing RA 3086

RA 3086



A4758-1(a) 500X Bright Light

Figure 3. Example of Crack on Inside of Drawn Tube

Draw Test 99D
Cumulative Reduction: 26.2% in four passes
Estimated Depth of Deepest Crack: 2 mils

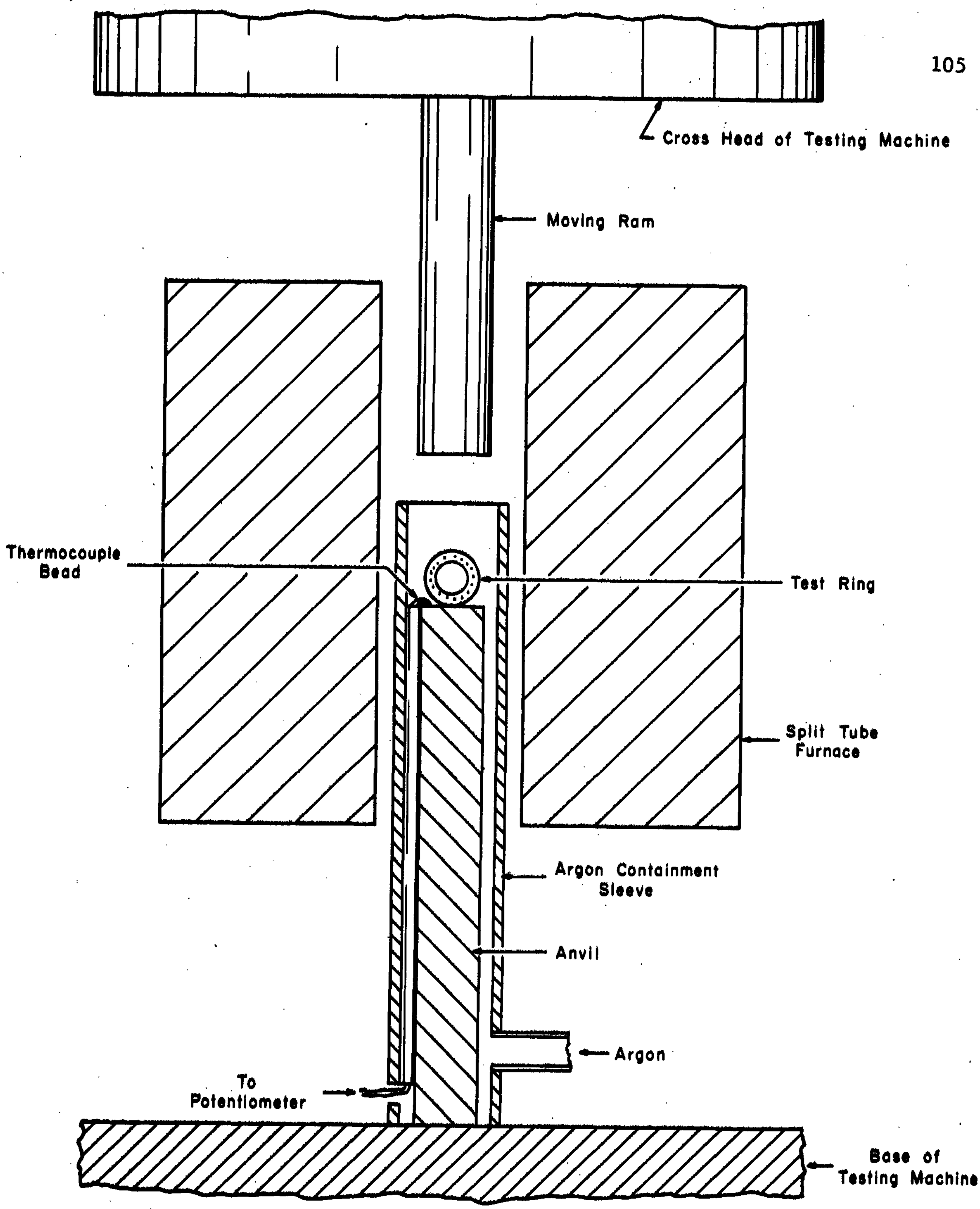
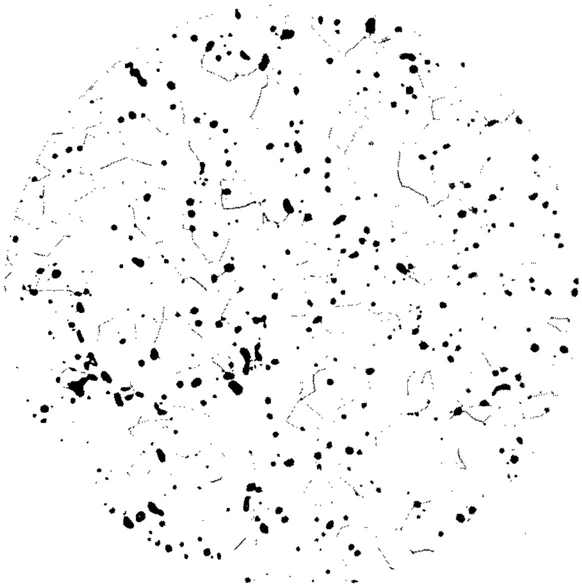
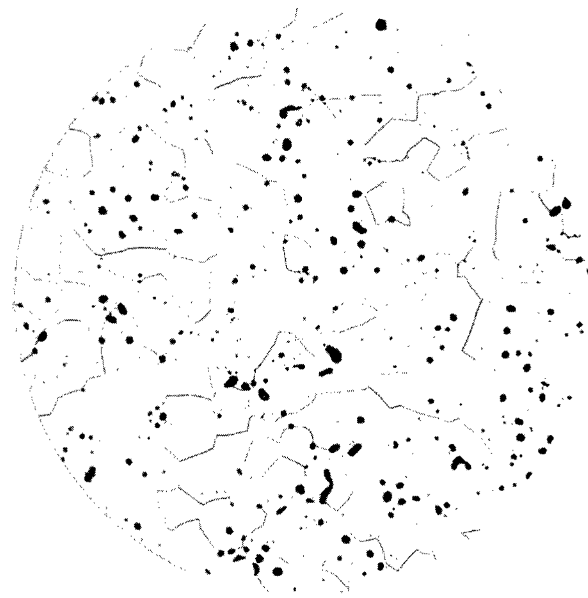


Figure 4. Ring Compression Test Assembly RA 2626



A 4785-1

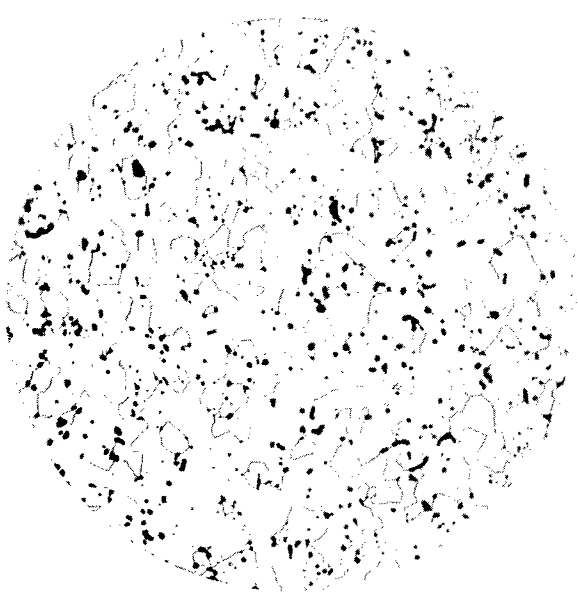
(a) Sleeve 214
97.4% T.D.



A 502 -22

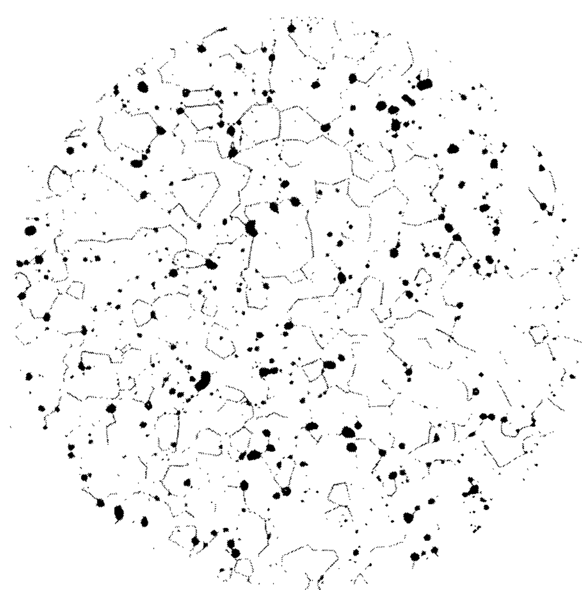
(b) Sleeve 235
97.4% T.D.

Bureau of Mines



A 4752-3

(c) Sleeve 210
96.3% T.D.

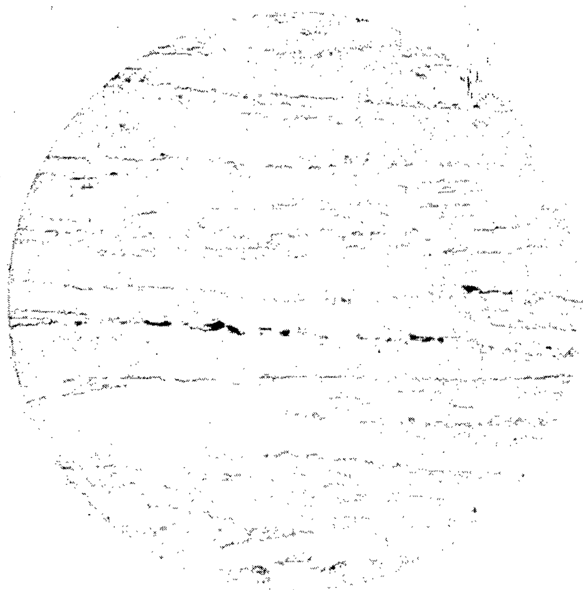


A 5017-7

(d) Sleeve 225
95.2% T.D.

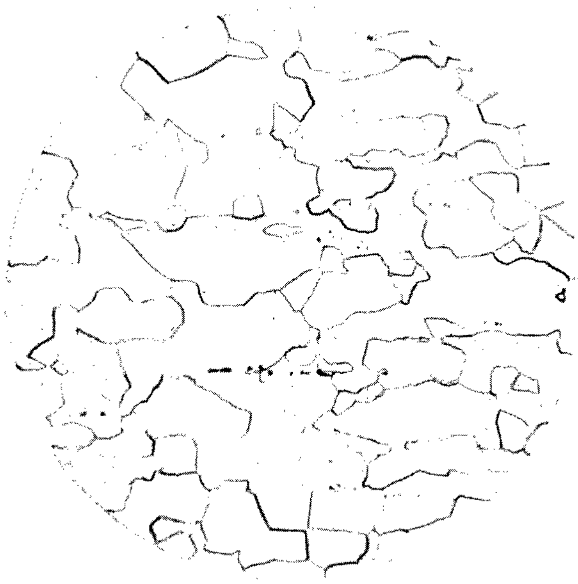
Vendor A

Figure 5: Microstructures of sintered W-25 $^{W}/_{o}$ Re extrusion sleeves. Attack-polished and etched with Murakami's reagent.
150X Bright Light



A 4777-14

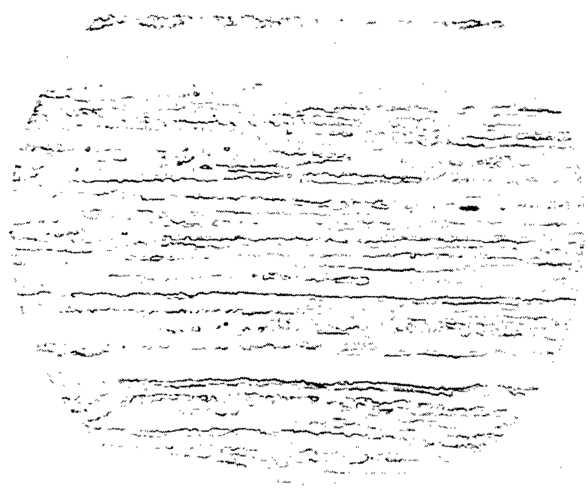
(a) As-extruded



A 4777-15

(b) Vacuum annealed 1 hr. at 1800°C

Figure 6. Microstructures of Tube 207 (rear). Attack-polished and etched with Murakami's reagent. 500X Bright Light



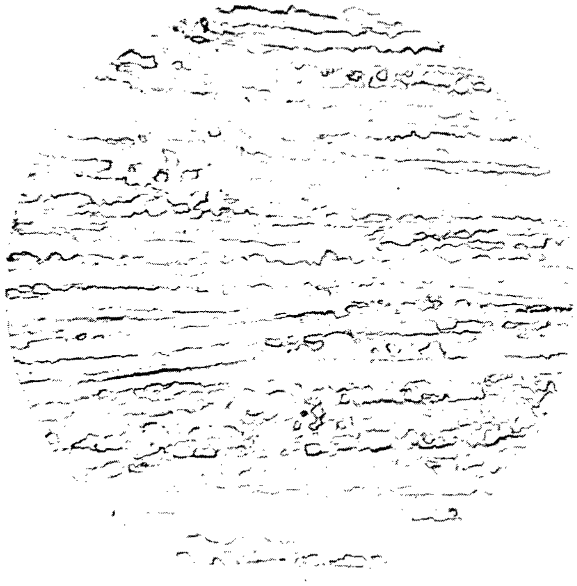
A 4777-24

(a) As-extruded

A 4777-25

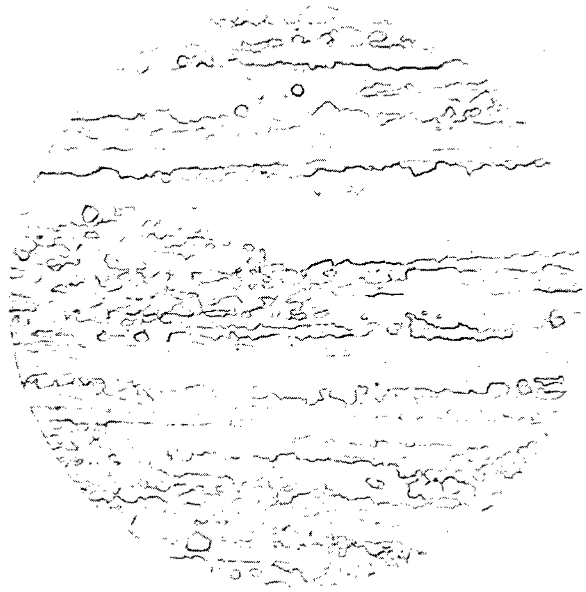
(b) Vacuum annealed 1 hr. at 1800^{o}C

Figure 7. Microstructures of Tube 211 (front). Attack-polished and etched with Murakami's reagent. 500X Bright Light



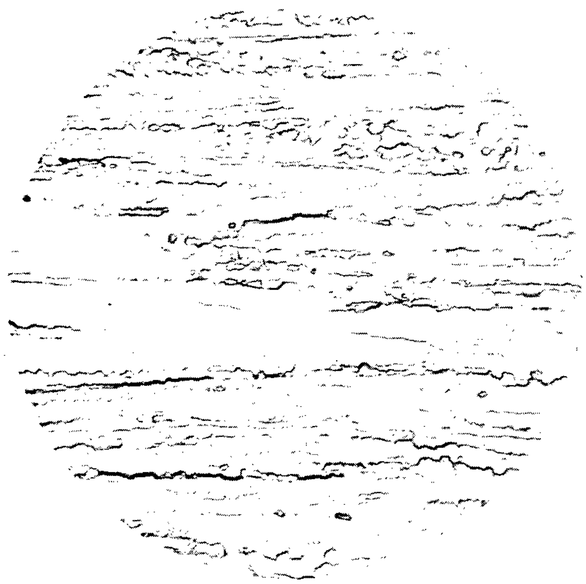
A 4999-4

(a) 4-3/4-inches from front



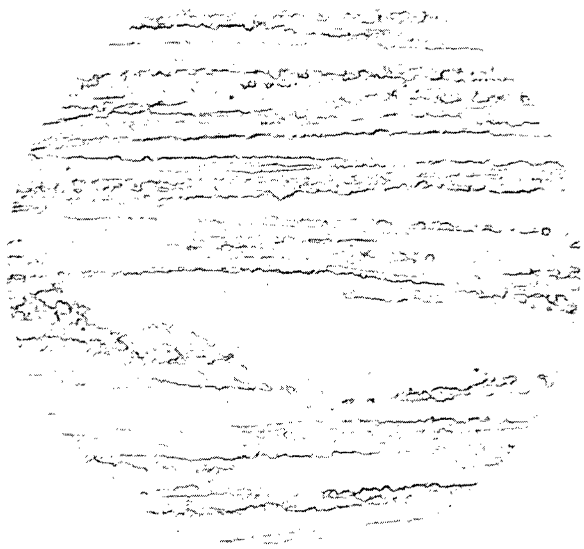
A 4999-5

(b) 36-inches from front



A 4999-6

(c) 68-inches from front



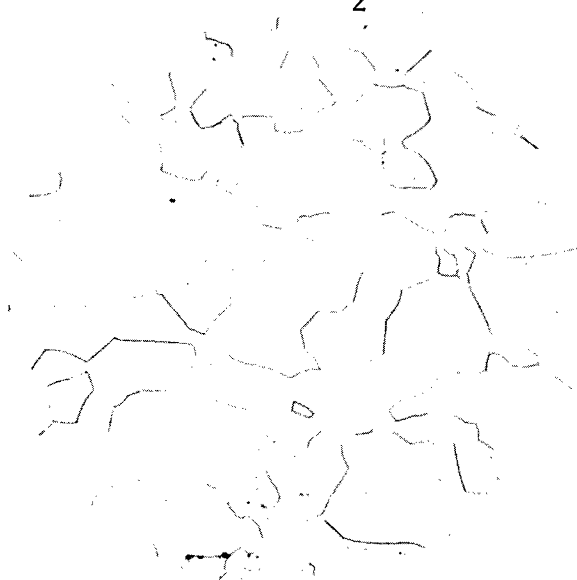
A 4999-7

(d) 100-inches from front

Figure 8. Microstructures of Tube 226 extruded at 1600°C with a 16:1 reduction. Attack-polished and etched with Murakami's reagent. 500X Bright Light

A4775-5 (a) Tube 198

Annealed 15 min. at 1800°C in A-15 H_2.

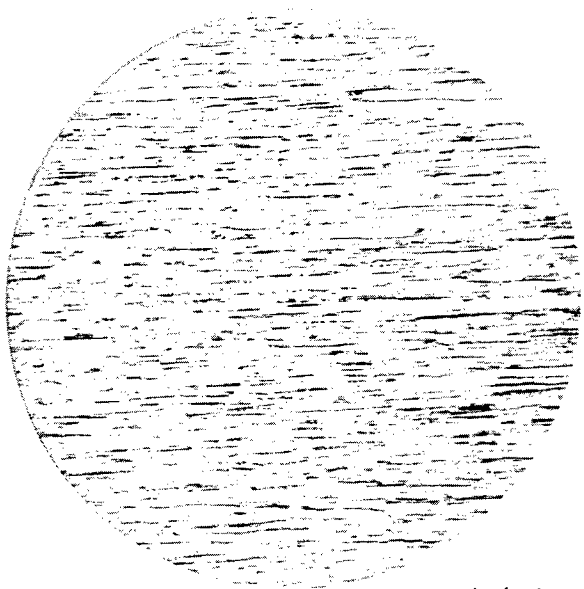


A4777-23 (b) Tube 199

Annealed 1 hr. at 1800°C in vacuum.

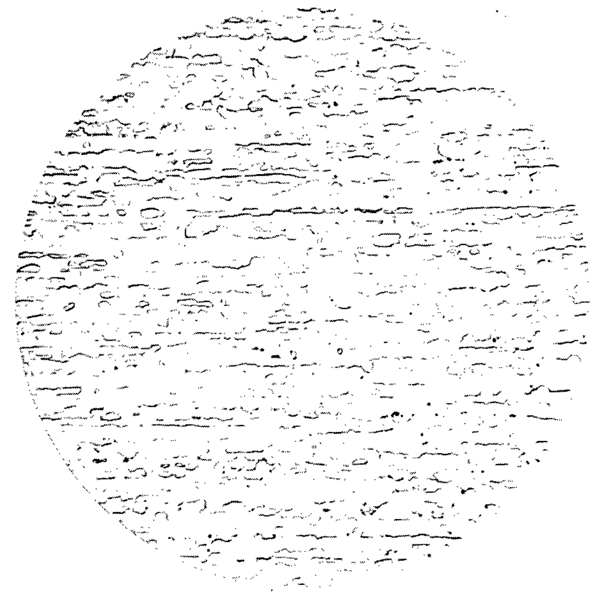
Figure 9. Microstructures of Annealed W - 25 w/o Re Tubes 198 and 199. Attack-polished and etched with Murakami's reagent.

500X Bright Light



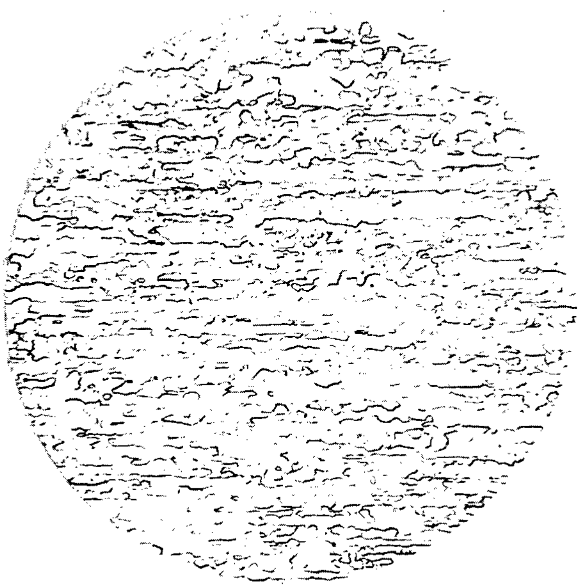
A 4729-9

(a) As-extruded



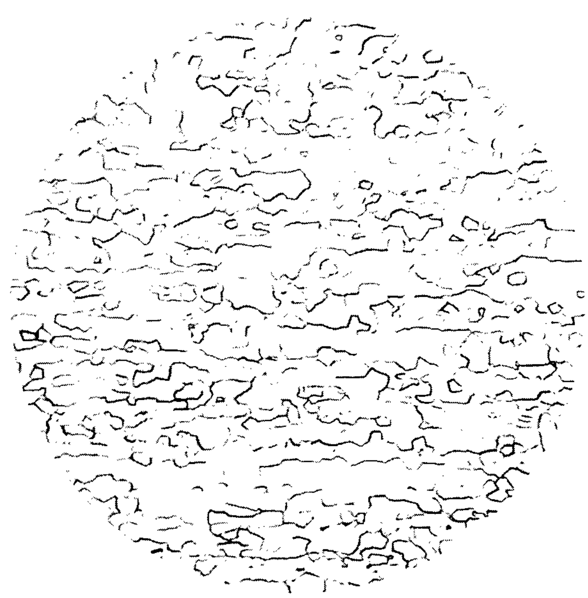
A 4729-10

(b) 1 hr. at 1400°C



A 4729-11

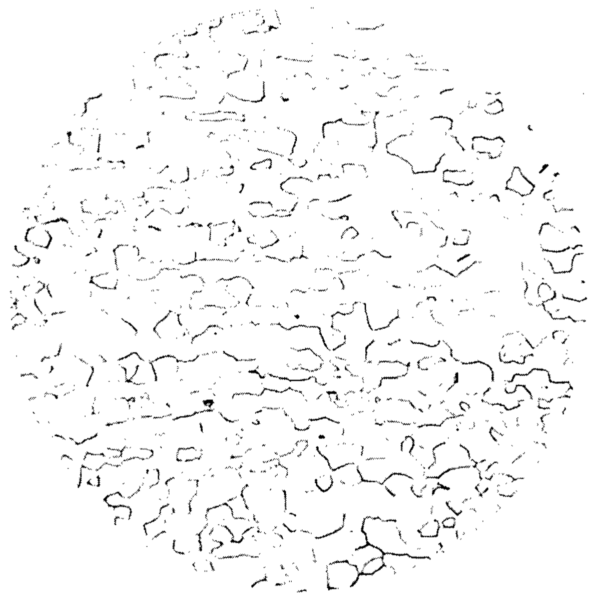
(c) 1 hr. at 1500°C



A 4729-12

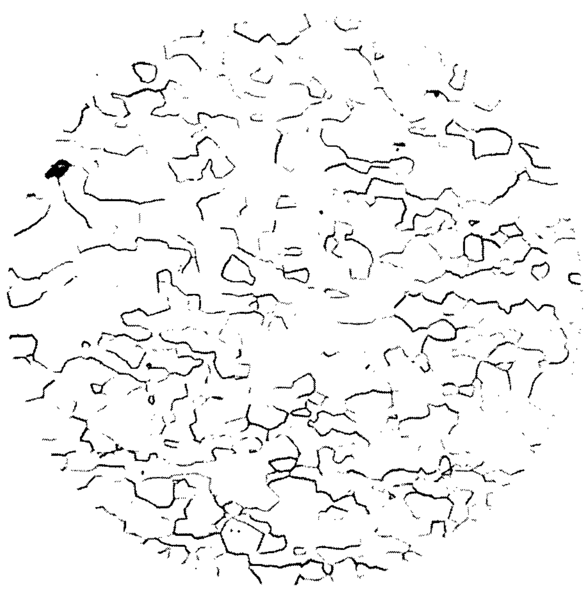
(d) 1 hr. at 1800°C

Figure 10. Microstructures of Tube 182 re-extruded at 1200°C with 25:1 reduction. Attack-polished and etched with Murakami's reagent. 500X Bright Light



A 4729-13

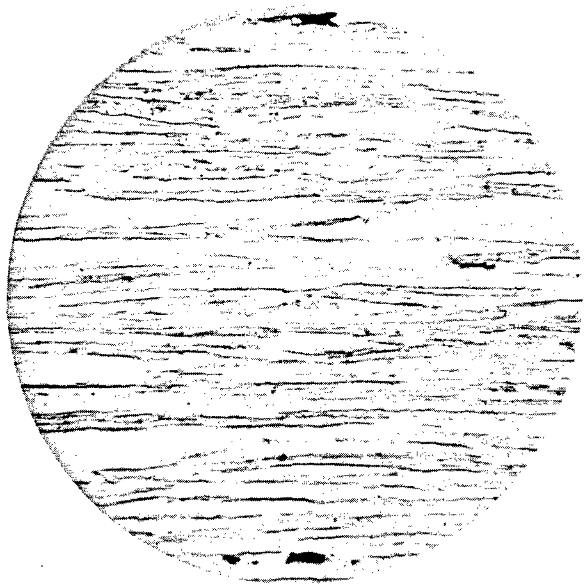
(e) 1 hr. 1700°C



A 4729-14

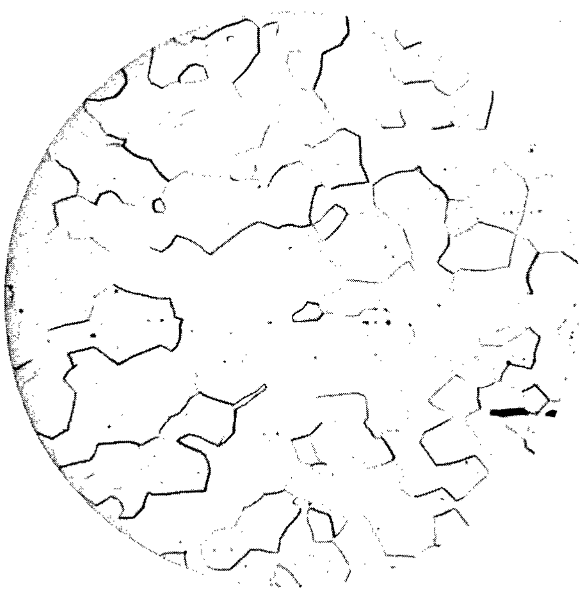
(f) 1 hr. 1800°C

Figure 10. (continued)



A 4792-1

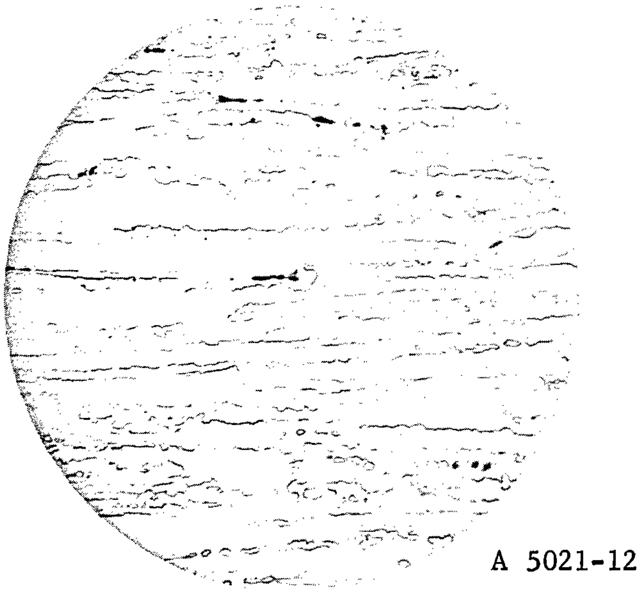
(a) As-extruded



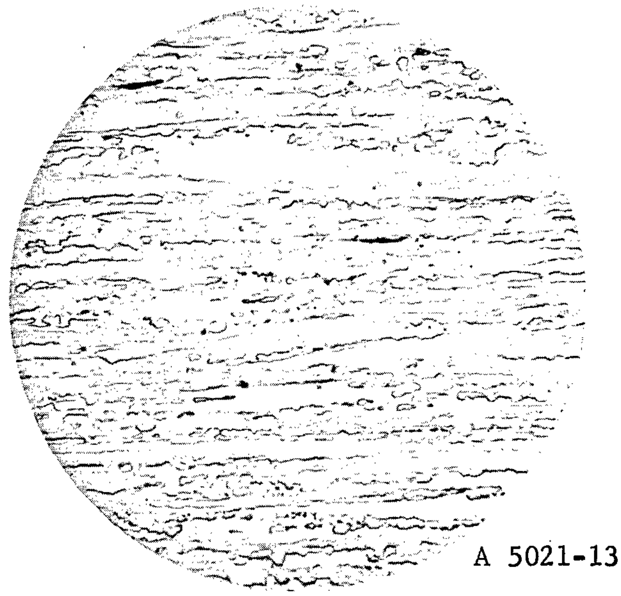
A 4999-2

(b) Vacuum annealed 1 hr. at 1800°C

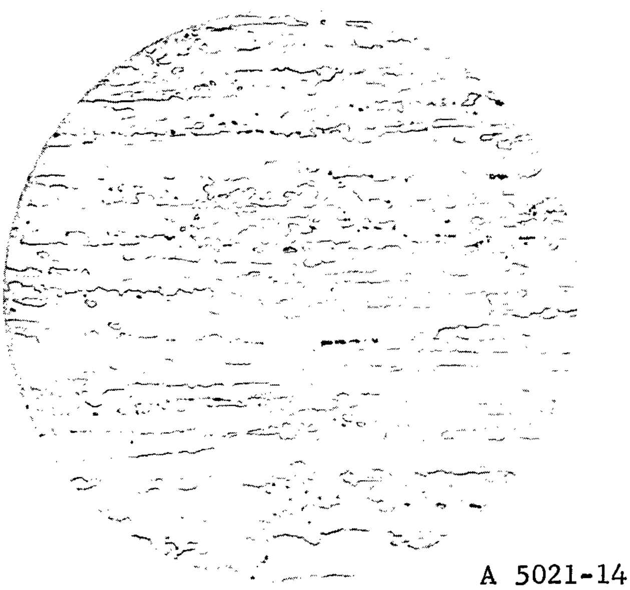
Figure 11.. Microstructures of Tube 223, re-extruded at 1200°C with 16:1 reduction. Attack-polished and etched with Murakami's reagent.
500X Bright Light



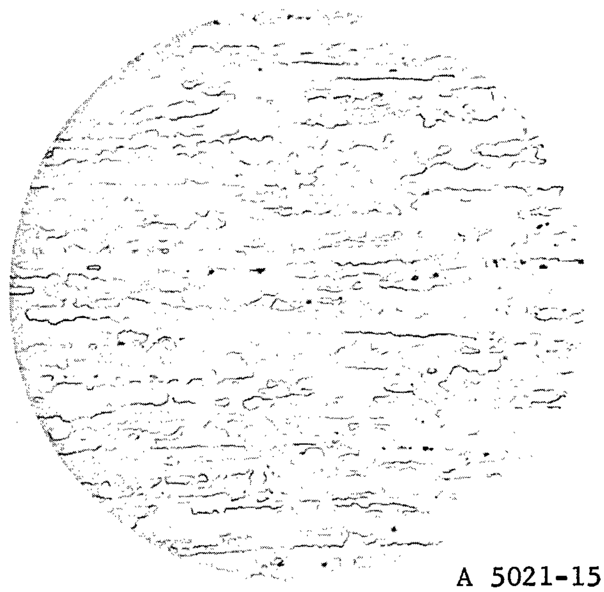
(a) Draw Test 166A
2.0% Cumulative Reduction



(b) Draw Test 166B
12.6% Cumulative Reduction



(c) Draw Test 166C
22.6% Cumulative Reduction



(d) Draw Test 166D
30.8% Cumulative Reduction

Figure 12. Microstructures of drawn re-extruded tubing. Tube 224 (1500°C re-extrusion) drawn at 510°C. Annealed 1/2 hour at 1400°C after each pass. Attack-polished and etched with Murakami's reagent. 500X Bright Light.

REFERENCES

1. J. G. Hunt, R. G. Jenkins, P. Loewenstein and A. M. White, "Development of a Fabrication Process for Seamless Tungsten Tubing", Summary Technical Report, October 17, 1963, NMI-1260.

2. S. Isserow, J. G. Hunt, R. G. Jenkins and A. L. Geary, "Fabrication of Seamless Tungsten and Tungsten-Rhenium Tubing II", Summary Technical Report, June 15, 1965, NMI-1264

3. R. N. Randall, H. F. Sawyer, "Development of Advanced Techniques for the Fabrication of Refractory Metal Tubing", Phase I Report, June 1, 1964 to October 31, 1965, NMI-9705.5.

APPENDIX I

PROCESS SEQUENCE FOR DRAWING 1/4-20 TUBING FROM 0.29-INCH OD x 30-MIL STOCK

(Cleared of molybdenum extrusion fillers)

(1) Stopper one end, fill tube with H_2O_2 (30 percent) for 1/2 hour, then rinse with water.

(2) Centerless polish outer surface with 320-grit silicon carbide belt; remove about 2 mils from OD.

(3) Electropolish outer and inner surface; remove about 1 mil layer from each (see p. 20).

(4) Point tube by swaging one end over TZM insert at 1400°C with chopper dies.

(5) Oxidize surface by rapidly heating in air to about 800°C for 5 to 10 seconds, then cooling.

(6) Apply graphite-sugar-alcohol slurry to the outer surface.

(7) Place tube on mandrel (M-2 or M-3 steel, R_c 60-65, surface oxidized at 900°F) for moving-mandrel draw.

(8) Place tube and mandrel in preheated furnace at 450°C butted against die heated to 450°C so that W - 25 Re point protrudes through die into draw grips (see Figure 2).

(9) When thermocouple adjacent to tube indicates 450°C for about one minute, begin to draw at slowest speed; push mandrel manually into tube at start of draw until friction begins to pull mandrel through die with tube.

(10) Adjust speed to attain 30-40 inches per minute in a few seconds.

(11) When draw completed, reel tube 6 to 8 passes at 480°C (same heating technique as for drawing); rolls, at room temperature, are coated with any greasy hot work lubricant such as "Oildag". Roll setting should be previously established by a series trial for the particular size (p.24).

(12) Allow the reeled tube and mandrel combination to cool to room temperature, then pull the mandrel from the tube using the draw bench. To do this, grip the point of the tube in a set of jaws fastened to the die head and grip the exposed end of the mandrel using a set of screw-activated grips which can hold the hardened steel.

(13) Vapor blast the tube after first plugging the open end with a stopper or loose paper plug.

(14) Remove the plug and fill the interior of the tube with aqua regia for 15 minutes (to help clean the interior prior to annealing).

(15) Place the clean tube inside a larger molybdenum tube in a furnace at 1400°C for 1/2-hour in wet flowing A - 15 H_2.

(16) Move the tube to a cold zone of the furnace where it can cool under the protective atmosphere. (The furnace used in this program had a useful temperature zone of about 18 inches long. Tubes longer than 18 inches were heat-treated one-half at a time).

(17) Use this drawing tool schedule:

Pass	Die Dia. (in.)	Mandrel Dia. (in.)
A	.270	.225
B	.260	.218
C	.250	.210
D	.250	None

(18) After the final anneal following Pass D, remove the point and scrub the tube thoroughly with a paste of Ajax cleanser (a test tube brush was used on the interior) then rinse.

(19) Soak the clean tube for three minutes in fused sodium hydroxide at 450°C, and allow the tube to cool to room temperature.

(20) Wash the sodium hydroxide from the tube, then rinse with water and dry.